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Alkali Silica reaction under the influence of chloride based deicers

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ALKALI SILICA REACTION UNDER THE INFLUENCE OF CHLORIDE BASED DEICERS

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ABSTRACT

Highways form the backbone of transportation in United States. As a result, issues related to distress in pavements and use of chemicals on pavement forms one of the major focus areas of highway agencies. One of the forms of distress in concrete pavements is Alkali silica reaction (ASR). The principal objective of this research study is to understand the role of chloride-based deicing chemical in causing or accelerating ASR distress. Sodium chloride or rock salt is one of the oldest forms of deicers. Several research studies have been done on ASR under the influence of sodium chloride and calcium chloride. However, limited information is available on effects of potassium chloride and magnesium chloride deicers. This research study is based on laboratory evaluation to assess effects of chloride deicers on alkali silica reactivity. Variations in exposure conditions, cement and aggregate are introduced to understand the effects of deicer solutions on a wider spectrum. Mortar bar tests based on ASTM C 1260 tests were conducted, in which reactive fused silica and non-reactive natural sand were used as aggregates and two Type I cement with different alkali contents (low and high) were used. Tests were carried out at two different storage temperatures, 38°C and 80°C.

Findings from these tests show that 3% wt. solutions of sodium chloride and potassium chloride deicers can cause more expansion in mortar specimens containing fused silica, than even the standard 1 normal sodium hydroxide solution. Calcium chloride and magnesium chloride solutions have shown no effect on initiating or accelerating ASR in the same test specimens. Higher temperature accelerated the ASR distress, regardless of the source of alkali. The alkali content of cement itself did not have

a major influence on mortar bar expansion, particularly in the presence of deicing solutions. However, this is due to the ample supply of alkalis from the deicer soak solution in the test method. Formation of chloroaluminates or Friedel's salt has been observed and is believed to be the principal mechanism through which the chlorides in the deicer solutions are chemically bound. With removal of chlorides from the solution, alkali hydroxides are formed, thus inducing the ASR distress and the accompanied expansions in the test specimens. Even though such chloroaluminates were observed in mortar specimens exposed to calcium and magnesium chloride solutions, no accompanying expansion was observed in those test specimens.

DEDICATION

This thesis is dedicated to my mother- Rekha Desai, my father- Rashes Desai, my Late Mother-in-law- Vandana Rajani, my Father-in-law- Jairamdas Rajani and my husband- Umesh Rajani. Their blessings and good wishes have always motivated me through my research tenure.

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CHAPTER ONE

INTRODUCTION

1.1 Need for the Research

Distress in concrete due to alkali silica reaction (ASR) is a well known problem in more than 50 countries across the world [1]. It is commonly visible as map-cracking on concrete surfaces. Cracks in concrete lead to loss of serviceability of the structure. Fundamentally, the source of this problem is the dissolution of amorphous, poorly crystalline silica in high pH solution.

A common environment formed by reactive aggregate, adequate alkalis and sufficient moisture can trigger alkali silica reaction in concrete. Deicers are a potential source of alkalis. There are several published research literature and case studies of alkali silica distress in concrete due to deicing salts. These deicers can be chloride based or acetate based.

One of the oldest and widely accepted deicer – Rock salt is a chloride based deicer. Several researchers have studied the potential of sodium chloride (commonly known as Rock salt) and calcium chloride to cause alkali silica reaction in concrete. Limited research has been done on ASR in presence of potassium chloride and magnesium chloride. Very few researchers have studied this effect in totality, i.e. considering the influence of parameters like temperature, aggregate and alkalis (from internal and external sources).

This research focuses on identifying the potential of chloride deicers to trigger ASR in concrete. It is supported by the study of underlying reaction mechanism through

which these deicers cause ASR in concrete. Research matrix is further expanded to understand the influence of temperature, type of cement (internal source of alkalis) and aggregate on alkali silica reaction in the presence of chloride deicers (external source of alkalis). The knowledge acquired by this research can help establish better and effective conditions to form a more reliable and accelerated alternate test method.

1.2 Research Objectives

The key objectives focused in this research are:

1. To compare the effect of various commercially available chloride based deicers on alkali silica reaction(ASR) in concrete
2. To study the potential chemical reactions by which chloride deicers cause ASR in concrete
3. To study the effect of variables like temperature, type of cement and aggregate on alkali silica reaction in mortar specimens subjected to different chloride based deicers

1.3 Scope of Research

Scope of this research study was limited by using selected materials, standard and modified test methods, wet chemistry and microscopy to accomplish the research objectives. Materials were selected with a view to introduce variables like type of cement and type of aggregate in the tests.

A potentially non-reactive aggregate and a potentially reactive aggregate were used. Aggregates used were Natural sand (non-reactive aggregate) and Fused silica (reactive aggregate). The reactivity of aggregates was predicted based on their established history and performance in previous research. Standard ASTM C 1260 test was

performed to confirm the potential reactivity of aggregates. ASR is caused by reactive aggregates only. Thus, performance of deicers in presence of non-reactive aggregate would serve as base line for estimating their potential to cause ASR while using reactive aggregates. Cement used was Lehigh Type I High alkali cement and Lafarge Type I Low alkali cement.

Titrations were performed using phenolphthalein as an indicator to estimate concentration of hydroxyl ions in deicer solution exposed to crushed mortar for a defined period. Inductively Coupled Plasma (ICP) test was done to estimate concentration of cations and anions in the same solution. X-ray diffraction technique was used to identify compounds formed as a result of interaction of deicers with cement hydration products. Scanning Electron microscopy (SEM) and Energy Dispersive X-ray (EDX) analysis techniques were used to carry out microstructure level study of the mortar specimens. Specimens for SEM analysis were prepared by manual hand polishing. The microstructure level study was to be executed at Clemson Electron Microscope facility using Hitachi S-3400 SEM.

Reagent grade and commercially used chloride salts will be referred to as ‘Deicer’ in this research. Four different commonly used chloride based deicer were used for this study. Three out of four deicers were reagent grade chemicals available in amorphous state. The fourth deicer used for the test was commercially available. Water was used as a soak solution to predict contribution of reactive aggregate and cement to cause alkali silica reaction. 1N sodium hydroxide soak solution results would serve as base line for

comparative performance of the deicers. Test matrix also involved use of sodium chloride and sodium hydroxide deicer in blended form.

1.4 Research Approach

This research was executed using following approach:

1. Perform literature review on previous research done in the area of alkali silica reaction and effect of chloride deicers on alkali silica reaction
2. Select aggregates based on their established history and performance in previous research in terms of reactivity to alkali silica reaction
3. Select concentration of deicer solution based on published literature
4. Prepare test matrix taking into account all the variables involved like type of aggregate, cement, deicer solution and temperature. Plan and schedule casting of mortar specimens based on the test matrix
5. Review of Standard ASTM C 1260 test, SEM/EDX analysis, titrations, ICP test and X-ray diffraction procedures
6. Prepare, monitor and study the mortar specimens in line with the research objectives

CHAPTER TWO

LITERATURE REVIEW

2.1 General

This chapter incorporates information from published literature on alkali silica reaction and deicers. It introduces alkali silica reaction distress in concrete, environment causing the distress, basic reaction mechanism and deicers. It further deals with how chloride based deicers cause alkali silica reaction in concrete.

2.2 Alkali silica reaction

Distress in concrete structures due to alkali aggregate reactions (AAR) was first observed in California, United States during the 1940s [1]. One of the more widespread forms of alkali aggregate reaction is alkali silica reaction. An alkali-silica reactive aggregate reacts with alkali hydroxides in the system to form alkali-silicate gel at the paste-aggregate interface. In the presence of moisture, the alkali silicate gel expands. Micro-cracks develop due to the restraint offered by concrete to the expanding gel. This expansive reaction is known as alkali silica reaction (ASR).

It is visually identified in concrete structures as random or map-cracking, patterned cracking oriented in the direction of least restraint, gel lining the cracks, spalling of joint, pop-outs over the reactive aggregates [3]. ASR in concrete is caused by inherent properties of the paste and thus the distress is not localized.

2.2.1 Environment causing alkali silica reaction

Environment formed by reactive silica, adequate concentration of alkali hydroxides (Na^+, K^+ and OH^-) in pore solution and sufficient moisture is an ideal environment for occurrence of alkali silica reaction.

1. Reactive silica

Reactivity of silica depends on aggregate mineralogy, degree of crystallinity and its solubility in pore solution. Contribution of a reactive aggregate towards ASR effect depends on:

a) Crystal structure of aggregate

In case of well crystallized and dense silica (like quartz) the attack by alkali and hydroxyl ions mainly takes place on the surface. If the crystal structure of silica is disordered then the number of reaction sites for alkali and hydroxyl ions to react with silica are increased. As a result, silica with a disordered/ unstable crystal structure (like opal) and increased solubility in highly alkaline solutions is most susceptible to alkali silica reaction. This is illustrated in Figure below. [4].

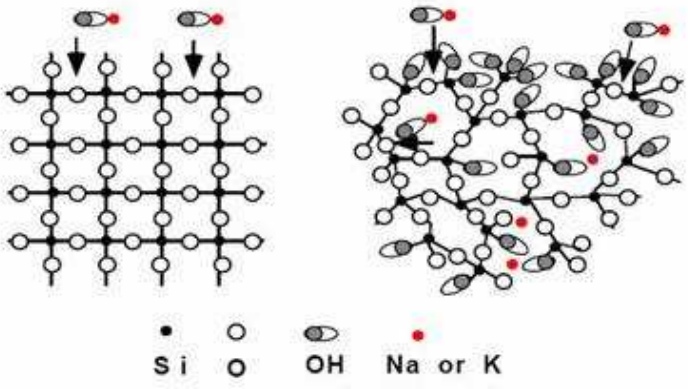


Figure 2.1: Crystal structure of Quartz (left) and Opal (right)

b) Proportion of reactive aggregate

As per recent research by Ichikawa [5], use of reactive aggregate beyond the pessimum proportion can reduce the ASR effect. This can be attributed to the decrease in calcium hydroxide and alkali hydroxide available per aggregate particle.

c) Particle size of reactive aggregate

Research by Ichikawa [5] concludes that for a fixed volume of reactive aggregate in a mix, increase in particle size increases ASR expansion but reduces the rate of ASR. Also, very fine reactive aggregate sizes tend to mitigate ASR by pozzolanic effect.

Table below shows a list of potential reactive minerals and rocks.

Table 2.1: Potentially alkali-silica reactive mineral phases and rocks (from CSA 1994a) [1]

A-1. Alkali-reactive, poorly crystalline, or metastable silica minerals and volcanic or artificial glasses (classical alkali–silica reaction)	
Reactants	Opal, tridymite, cristobalite Acidic, intermediate, and basic volcanic glass Artificial glass (e.g., common glass, steel slag, pyrex), beekite
Rocks	Sedimentary rocks containing opal, such as shale, sandstone, silicified rock, some chert and flint, and diatomite Glassy to vitrophyric volcanic rocks: acidic, intermediate, and basic, such as rhyolite, dacite, latite, andesite and their tuff, perlite, obsidian, all varieties with a glassy groundmass, some basalt
A-2. Alkali-reactive quartz-bearing rocks	
Reactants	Chalcedony, cryptocrystalline, microcrystalline quartz Macrogranular quartz With deformed crystal lattice Rich in inclusions Intensively fractured or granulated With microcrystalline quartz at grain boundaries
Rocks	Chert, flint, quartz vein, quartzite, quartzarenite, quartzitic sandstone, siliceous limestone Volcanic rocks such as in A1, but with devitrified, cryptocrystalline to microcrystalline groundmass Microgranular to macrogranular rocks of various origins: contain microcrystalline to cryptocrystalline quartz, or significant amounts of moderately to highly strained quartz: Igneous: granite, granodiorite, charnockite Sedimentary: sandstone, greywacke, siltstone, shale, siliceous limestone, arenite, arkose Metamorphic: gneiss, quartz-mica schist, quartzite, hornfel, phyllite argillite, slate

2. Alkalis in Cement and in Concrete pore solution

Alkalis refer to the elements listed in the first column of the periodic table. Two of the alkalis that occur commonly in cement are sodium and potassium. Sources of alkali in concrete pore solution are alkali sulfates in cement, mix water, supplementary cementitious materials and deicing salts. It is predicted based on experiments that alkalis in the form of sodium and potassium are adsorbed and do not chemically form the structure of alkali silica gel.[8]

Total alkali content is expressed as Na_2O equivalent rather than the individual concentrations of alkali oxides [6]

Equivalent Alkalies, $Na_2O = Na_2O + 0.658 \times K_2O$

Alkali silica reaction is caused by action of hydroxyl ions, and not alkalis, on reactive aggregate [6]. This paper also refers to past literature which state that ASR and physical characteristics of ASR products are affected by concentration of alkalis in the paste although they are not the cause of the reaction. Recent research study by Leeman and Lothenbach [7] conclude that ASR expansion is influenced by K/Na ratio. Pore solution results in this study indicated that the difference in expansion was attributed to composition of alkali cations in solution and not hydroxyl ion concentration.

3. Hydroxyl ion concentration

Alkali cations in cement will produce an equivalent number of hydroxide ions only after the formation of ettringite. In case of specimens subjected to alkali salt solution, alkali hydroxides formed as a result represent the external source of hydroxyl ions.

4. Sufficient Moisture

Research findings show that alkali silica reaction in concrete occurs when relative humidity is greater than 80-85% [1]. Moisture in the system facilitates the migration of alkali ions to reaction sites and thus accelerates the alkali silica reaction. Alkali silica gel swells by absorbing moisture. This results in micro-cracking due to expansive stresses on concrete.

2.2.1 Understanding of Alkali Silica reaction at a micro-level

Previous research studies on alkali silica reaction conclude that the process of alkali silica reaction involves alkali cations, calcium ions, hydroxyl ions and reactive silica phases in aggregate [8-13].

The alkali silica reaction can be well understood based on reaction mechanism proposed by Ichikawa [5,13] and source of expansive pressure exerted by ASR gel.[14]

a) Alkalis and hydroxyl ions break the siloxane networks in aggregate particle to form alkali silicate gel.

$$\equiv Si\text{-}O\text{-}Si \equiv + R^{+} + OH^{-} \rightarrow \equiv Si\text{-}O\text{-}R + H\text{-}O\text{-}Si \equiv$$

$$R^{+} + OH^{-} + H\text{-}O\text{-}Si \equiv \rightarrow \equiv Si\text{-}O\text{-}R + H_2O$$

In presence of moisture this gel forms hydrated alkali silicate gel. As reaction progresses towards the center of aggregate particle, a highly hydrated mature alkali silicate gel overlies a less hydrated immature alkali silicate gel. Part of the mature alkali silicate gel migrates into pore solution.

b) Dissolution of calcium ions and formation of calcium alkali silicate gel

Calcium hydroxide, a cement hydration product, forms the primary source of Ca^{+2} and OH^- ions. As hydroxyl ions are used in the reaction, calcium ions dissolve in the pore solution. Mature alkali silicate gel in the pore solution and around the aggregate reacts with Ca^{+2} ions to form fragmental calcium alkali silicate gel (high Ca/Si ratio).

$$\equiv Si\text{-}O\text{-}R + Ca^{+2} + R\text{-}O\text{-}Si \equiv \rightarrow \equiv Si\text{-}O\text{-}Ca^{+2}\text{-}O\text{-}Si \equiv + 2R^+$$

The inner immature less hydrated alkali silicate gel react with the slowly diffusing Ca^{+2} ions to form a hard rim of calcium alkali silicate (low Ca/Si ratio). Released alkalis further propagate the reaction. The newly formed rim allows flow of alkalis and hydroxyl ions. It confines the alkali silicate gel within its boundary which results in build up of expansive pressures. This ultimately results in cracking of aggregate.

Several other researches in this field also propose a similar mechanism for alkali silica reaction. [9,11]

Diffusion of calcium ions to the reaction site is slow in comparison to the alkalis. As per H. Wang [11], this can be due to the increased solubility of alkali hydroxides in comparison to the calcium hydroxide. This makes them readily available at the reaction sites. Also, smaller hydraulic radius of Na^+ and K^+ ions in comparison to Ca^{+2} ions increases their rate of diffusion. Research studies have shown that Ca^{+2} ions are vital for alkali silica reaction to initiate and propagate in concrete [8,10-12]. As per hypothesis by Powers [9], calcium alkali silicate gel which has more Ca^{+2} ions adsorbed on reactive silica compared to alkalis is non-expansive in nature.

c) Expansive pressure resulting exerted by ASR gel

As per research work done by Prezzi [14], expansion of alkali silica gel can be well explained by the double layer theory. Double layer is composed of Na^{+}, K^{+} and Ca^{+2} ions. It balances the negative charge on silica grain. Reactions involved in the formation of double layer are,

$\equiv Si\text{-}O\text{-}Si \equiv + R^{+} + OH^{-} \rightarrow \equiv Si\text{-}O\text{-}R + H\text{-}O\text{-}Si \equiv$

$R^{+} + OH^{-} + H\text{-}O\text{-}Si \equiv \rightarrow \equiv Si\text{-}O\text{-}R + H_2O$

Thickness of double layer increases with the decrease in valence of ions and concentration of electrolyte. In case of mortar specimens subjected to air, double layer thickness reduces due to the increased concentration of ions. As a result, Van der Waals force exceeds the repulsive force between ions and gel is formed. When mortar specimens are subjected to water/solution, the gel absorbs water. With decrease in concentration of solution at the reaction site the double layer thickness increases. Repulsive forces between ions increases, gel expands and exerts pressure on concrete. Cracks occur when expansive pressure exceeds the tensile capacity of concrete.[9,15]

2.3 Deicers

2.3.1 Introduction

Deicing chemicals have been extensively used since 1950s to clear highways of snow/ice accumulation during winter. Increasing dependence on motor vehicles for transportation makes these chemicals one of the major focus areas of highway agencies transportation.

2.3.2 Action of Deicers

Deicers melt on coming in contact with ice and form brine (salty solution). Brine has a freezing point below 32°F (0°C). As a result, brine penetrates and spreads through the layer of ice thereby weakening the pavement-ice bond [16].

2.4 Commonly used deicers for highways [17,18]

2.4.1 Chloride based deicers

Commonly used chloride based deicers are sodium chloride (rock salt), calcium chloride and magnesium chloride.

1. Sodium chloride (Rock salt)

It is the oldest and most commonly used highway deicer. Sodium chloride is primarily used in solid form. At a concentration of 23.3%, sodium chloride freezes at a minimum temperature of -5.8°F (-21°C).

2. Calcium chloride

This deicer can be used in the form of pellets, flake or liquid. The freezing point of calcium chloride, -60°F (-51°C) at 29.8% concentration, is lower than that of sodium chloride. This makes it more effective in extremely cold regions. Key aspects that limit its use are high cost and high corrosivity. It is obtained from natural well brines through Solvay process. Calcium chloride and Sodium carbonate are by-products of Solvay process that involves blending sodium chloride and calcium carbonate.

3. Magnesium chloride

Magnesium chloride is derived from natural brines. It has a freezing point of -28°F (-33°C) at 21.6% concentration.

2.4.2 Acetate based deicers

These deicers involve use of highly concentrated acetic acid as a reactant. Thus, use of these deicers for large scale application may require attention.

1. Calcium magnesium acetate (CMA)

This deicer was developed during the 1970s with the support of FHWA. The freezing point of CMA deicer is -17.5°F (-27.5°C) at 32.5% concentration. Some of the properties that restrict its use as a deicer are slow action, less effective at lower temperatures, low density and high volume requirement. CMA deicer is less corrosive to reinforcing steel.

2. Potassium acetate

Potassium acetate is used in liquid form only. It has a freezing point of -76°F (-60°C) at 49% concentration. It is a non-chloride deicer.

2.4.3 Glycol based deicers

Use of glycol based compounds like ethylene glycols and propylene glycols are used as aircraft deicers. They have not been used as highway deicers.

2.4.4 Urea

Urea is a soluble nitrogenous compound and a weak base. It has low corrosion potential. It has limited use due to its degradation to ammonia which is detrimental to aquatic life in streams running near highways.

2.5 Effect of Chloride based deicers on Alkali silica reaction in concrete

It is well known that deicers involving chlorides can result in corrosion of reinforcement in concrete. Chlorides in concrete can be found in free state or bound with

hydration products. Free chloride levels may be affected by type of cations and chloride dosage [19]. Several research studies have shown that alkali chlorides seem to trigger the alkali silica reaction in concrete. Mortar specimens have been subjected to alkali chlorides by introducing them as additives in the mix or by using external alkali salt solution.

Alkali salts seem to cause more expansion in concrete in comparison to alkali hydroxides [2]. Monovalent cations (e.g. Na^{+}, K^{+}) have a higher expansion potential compared to divalent or trivalent cations (e.g. Ca^{+2}) [20]. Exposure to chloride solutions leads to the formation of chloroaluminates [21].

Behavior of mortar specimens subjected to the chloride deicers depends on the concentration of deicing solution and exposure to conditions like temperature.

1. Sodium chloride

Extensive research has been done on effect of sodium chloride on alkali silica reaction. SO_4^{-2} ions from ettringite are replaced by Cl^{-} from the sodium chloride solution leading to the formation of chloroaluminates.

$2\ NaCl + Ca(OH)_2 \rightarrow 2\ NaOH + CaCl_2$

$3CaO.Al_2O_3.3CaSO_4.32H_2O + CaCl_2 \rightarrow 3CaO.Al_2O_3.3CaCl_2.32H_2O + 3CaSO_4$

Sodium chloride tends to increase the hydroxyl ion concentration in pore solution [22,23]. This may be due to the formation of chloride intruded ASR products.

Berube [24] suggests that in mortar specimens exposed to sodium chloride deicing solution, soluble Cl^{-} ions may be present as sodium chloride and total chlorides

may be present as calcium chloroaluminate. The paper also cites reference which states that the transformation of soluble chloride ions to chloroaluminates is a timely process.

Pore solution of mortar samples involving reactive aggregate and sodium chloride as additive showed slight decrease in OH^- ion concentration and a drastic reduction in Cl^- ion concentration between the age of 12-24 hours. Pore solution of mortar samples involving a non-reactive aggregate and sodium chloride as additive showed little drop in the OH^- ion concentration and Cl^- ion concentration at the same age. Based on this study, Kawamura [22] concluded that rapid expansion in early stage of the alkali silica reaction can be due to chloride ions.

2. Calcium chloride

Presence of free calcium hydroxide and a high concentration of $CaCl_2$ deicer can cause failure of concrete [25]. Chloride concentration in mortar specimens may be due to formation of chloroaluminates ($3CaO.Al_2O_3.3CaCl_2.32H_2O$), calcium chloride hydrates ($3CaO.CaCl_2.12H_2O$, $3CaO.CaCl_2.2H_2O$) or adsorption of Cl^- by Calcium silicate hydrate (CSH) [21,25].

Mortar specimens subjected to $CaCl_2$ solution showed less expansion in comparison to mortar specimens subjected to NaCl deicer solution [20,22]. Expansion in such specimens may be attributed to Cl^- ions. This is supported by the fact that the hydroxyl ion concentration in pore solution involving a $CaCl_2$ environment was less than in pore solution involving a chloride free environment.

3. Potassium chloride

Potassium salt solutions seem to aggravate ASR more compared to sodium salt solutions [2]. Reaction mechanism is similar to that of sodium chloride.

4. Magnesium chloride

Magnesium chloride as a deicer does not contribute to alkali silica reaction. It causes distress in concrete by reaction with cementitious CSH to form non-cementitious Magnesium silicate hydrate.

CHAPTER THREE

MATERIALS

Materials used were in line with the research objectives. Aggregates used were Natural sand and Fused silica. Two different types of cement were used for preparing mortar specimens - Type I high alkali cement and Type I low alkali cement. Four different deicers were used for this study. The reagent grade chemicals used in amorphous and pure state were Sodium chloride, Calcium chloride and Potassium chloride. Commercially available and commonly used Magnesium chloride deicer was also included in the study. Other soak solutions used were 1N sodium hydroxide solution, de-ionized water and solution comprised of 0.5N Sodium hydroxide and 3% Sodium chloride.

3.1 Aggregates

A reactive aggregate and a non-reactive aggregate were used for this study.

1. Sand

Non-reactive aggregate used was natural sand. Sand was thoroughly washed and oven dried before use in the experiments. The sand was sieved and batched as per the gradation requirements of Standard ASTM C 1260 procedure.

2. Fused silica

Reactive aggregate was included in the test matrix as it contributes towards the environment required for ASR to occur. The aggregate, fused silica, used in the tests was a high purity synthetic amorphous silicon dioxide aggregate. Fused silica used was

uniformly graded. It was used to replace sand by an amount that equals 50 percent of the total aggregate weight used in the mix.

Table 3.1: Properties of Aggregates

Aggregate Properties	Natural sand	Fused silica
Bulk specific gravity (OD)	1.602	x
Bulk specific gravity (SSD)	1.922	x
% Water absorption	1.8	0.4

3.2 Cement

Type I high alkali cement and Type I low alkali cement were used for this research. Lehigh Type I High alkali cement has an equivalent alkali content of 0.82%. The source for this cement was Evansville, PA. Lafarge Type I Low alkali cement has an equivalent alkali content of 0.31%. The source for this cement was Harleyville, SC. Table below shows the chemical composition/ physical properties of cement used.

Table 3.2: Chemical Composition/ Physical Properties of Cement

Chemical/ Physical Properties (%)	Lehigh Type I High alkali cement	Lafarge Type I Low alkali cement
Silicon dioxide SiO_2	19.74	20.3
Aluminium oxide Al_2O_3	4.98	5.0
Ferric oxide Fe_2O_3	3.13	3.4
Calcium oxide CaO	61.84	64.45
Magnesium oxide MgO	2.54	1.2
Sulfur trioxide SO_3	4.15	2.8
Ignition loss	1.9	1.85
Equivalent alkalis (Na_2O eq.)	0.82	0.31
Insoluble residue	0.25	0.17
Tricalcium aluminate C_3A	8.97	7.2
Tricalcium silicate C_3S	46.6	63.76
Autoclave Expansion	0.12	0.08

3.3 Deicing/Reagent grade Chemicals

Deicing/ Reagent grade chemicals used were Sodium chloride, Calcium chloride, Potassium chloride and Magnesium chloride. Blend of sodium chloride and sodium chloride deicer solution was also used to study their combined effect on alkali silica reaction. 3% deicer solution was prepared for each deicer used, i.e. sodium chloride, calcium chloride, potassium chloride deicer and magnesium chloride.

A liter of 3% solution was prepared by mixing 30 gms of deicer in de-ionized water.

1. Sodium chloride

This reagent grade chemical was obtained in granular state from Fisher Scientific.

2. Calcium chloride

This reagent grade chemical was obtained in 96% extra pure, powder, anhydrous form from ACROS Organics.

3. Potassium chloride

Potassium chloride was obtained in extra pure, powder form from ACROS Organics as a reagent grade chemical.

4. Magnesium chloride

MAG (Nature's Total Ice Controller) was used. This is a commercially available and used deicer manufactured by Dead Sea Works Limited, Israel. This deicer is a blend of magnesium chloride, calcium chloride, potassium chloride and sodium chloride. Details of product contents are,

Calcium chloride (2-3%)

Potassium chloride (0.5-1%)

Sodium chloride (0.5-1%)

Magnesium chloride hexahydrate (Balance)

5. 0.5N sodium hydroxide and 3 % sodium chloride solution

The deicer solution involved blend of sodium chloride and sodium hydroxide. A liter of this blended deicer solution was prepared using following procedure:

Prepare 500 ml of 1N sodium hydroxide solution, add 30 grams of sodium chloride deicer and finally dilute the solution to one liter by adding de-ionized water

3.4 General Notation for Identification

Subject matter of thesis involves reference to materials used by means of notations. Table below indicates list of materials used and the notation used for their identification.

Table 3.3: List of Notations

Material	Notation
Sand	S
Fused silica	FS
Type I Low alkali cement	LC
Type I High alkali cement	HC
De-ionized water	0
3% sodium chloride (NaCl) deicer solution	1
3% calcium chloride($CaCl_2$) deicer solution	2
3% potassium chloride deicer(KCl) solution	3
1N sodium hydroxide(NaOH) solution	4
Blended deicer(1N NaOH+3% NaCl) solution	5
3% magnesium chloride($MgCl_2$) deicer solution	6

CHAPTER FOUR

PROCEDURES

Standard ASTM C 1260 and Mortar bar Test methods were used to find percentage expansion in mortar bars subjected to different deicing solutions. Titrations, Inductively Coupled Plasma (ICP) test, Standard electron microscopy (SEM) and Energy dispersive X-ray (EDX), X-ray diffraction techniques were used to study the environment at microstructure level.

4.1 Standard ASTM C 1260

Standard ASTM C 1260 (also known as 'Accelerated Mortar Bar Test (AMBT)') was used to determine the potential of an aggregate to cause alkali silica reaction. Reactivity of aggregate was established by expansion greater than 0.1% after immersing the mortar bars in soak solution for 14 days.

Mortar mix was prepared using aggregates and cement as per test matrix of this research. Aggregates were graded as per the requirements mentioned in the procedure. Cement used was sieved through 850 μm (No. 20) sieve to avoid any lumps. Mortar specimens of the size (25 mm x 25 mm x 285 mm) were cast. Quantitative details of each ingredient of mortar mix to be prepared per batch are as below:

Aggregate to cement ratio: 2.25

Water- cement ratio: 0.47 % by mass

Water-cement ratio was adjusted based on the % water absorption of the aggregate. The mortar mix was prepared, mixed and cast into the moulds as per the requirements of the Standard. Mortar specimens in moulds were allowed to cure for 24 hrs and then removed

from the moulds. Removed specimens were immersed in tap water and were placed in the oven at 80°C for 24 hrs. Zero-day reading was noted at the end of 24 hrs and specimens were subjected to 1N Sodium hydroxide solution at 80°C. Ratio of volume of solution to volume of mortar specimens was considered as 4.0.

Each batch of mortar mix was used to prepare 3 mortar specimens. Expansion results of mortar specimens subjected to 1N Sodium hydroxide solution were recorded for 28 days at frequent intervals. Average % expansion results were used to interpret the test results.

4.2 Mortar Bar Test

The Mortar bar test is similar in aspects like preparing mix, casting and recording expansion results to the Standard ASTM C 1260 procedure. Temperature and deicing solutions to which the mortar specimens were exposed differentiates the Mortar bar test from the Standard ASTM C 1260 procedure. The following conditions were adopted in this procedure,

1. Mortar specimens at 38°C were subjected to 1N Sodium hydroxide solution, de-ionized water, 3 % Sodium chloride solution, 3 % Calcium chloride solution, 3% Potassium chloride solution, 0.5N Sodium hydroxide and 3% Sodium chloride as blended solution and 3% Magnesium chloride solution respectively.
2. Mortar specimens at 80°C were subjected to de-ionized water, 3 % Sodium chloride solution, 3 % Calcium chloride solution, 3% Potassium chloride solution, 0.5N Sodium hydroxide and 3% Sodium chloride as blended solution and 3% Magnesium chloride solution respectively.

4.3 Titrations

Solutions in cement paste possess high ionic strength. This limits the mobility of hydrogen ions and in turn results in a low activity coefficient. This results in increased deviation from the actual value while calculating hydroxyl ion concentration using pH measurements [6][26]. Based on this understanding, hydroxyl ion concentrations are measured by titrations rather than pH measurement.

Mortar specimens were prepared using fused silica and sand as per Standard ASTM C 1260 procedure. The ratio, (fused silica: sand) was kept same as that used in preparing mortar specimens in Standard ASTM C 1260 and Mortar Bar test. The de-moulded specimens were then placed in the curing room at 38°C in a container. The container was filled with water such that it does not touch the mortar specimens. Mortar specimens were cured for 14 days. After 14 days the mortar bars were crushed and passed through #4 sieve. Aggregates retained on #8, #16, #30, #50 sieve were used for titrations. Finer fraction of aggregates was avoided to prevent loss of aggregates. Aggregates collected for titration were subjected to deicer solution for about 28 days. Concentration of deicing solution was kept same as in Standard ASTM C 1260 and Mortar Bar test. Ratio of volume of solution to volume of mortar specimens was considered as 4.0. Solution was then filtered from the aggregate and diluted in the ratio 1:25. The diluted solution was then titrated with 0.1N hydrochloric acid using phenolphthalein as an indicator. 2-3 drops of phenolphthalein indicator were added. Titration involved a stepwise addition of 0.1N hydrochloric acid until the solution became colorless.

Based on the titration results the molarity/ hydroxyl ion concentration in deicing solution exposed to crushed mortar were predicted.

4.4 Inductively Coupled Plasma (ICP) Test

Filtered solution obtained for titrations was also used for the Inductively Coupled Plasma (ICP) Test. The solution was stored in small plastic containers. The solutions were diluted in the ratio of 1:100 and then given for the test. The ICP test were conducted at Agricultural Service Laboratory, Clemson University. Also, ionic concentration of this solution was compared to the ionic concentration of deicer solutions used in the research program

4.5 Standard Electron Microscopy (SEM) and Energy Dispersive X- ray (EDX) analysis

Study of microstructure of mortar specimens using Standard electron microscope and obtaining elemental composition of area of interest using Energy dispersive X- ray analysis was executed at Clemson Electron Microscope facility using Hitachi S-3400 Scanning Electron microscope. The purpose of SEM/EDX study was to confirm that expansion is due to alkali silica reaction.

Sample preparation for SEM/EDX analysis involved three distinct stages- Epoxy encapsulation, Vacuum Impregnation and Preparation of polished specimens.

1. Epoxy Encapsulation

Specimens for SEM study were obtained from mortar bars using a mechanical saw. The sample was cleaned in de-ionized water to remove debri from the surface of sample caused as a result of sawing. Sample was allowed to dry completely. This is important for effective polymerization and adhesion of the epoxy. Epoxy resin

encapsulation improves external and internal integrity of the sample. Allied EpoxySet kit (a product of Allied High Tech Products, Inc.) was used for this process.

The set includes the resin and hardener. Resin and hardener were mixed in a ratio of 100: 12 as per manufacturer's specifications. Encapsulation involved the immersion of sample in epoxy resin in a mould. All samples were identified by labels inserted during the immersion.

2. Vacuum Impregnation

Mould with sample immersed in epoxy resin was then subjected to the Allied Vacuum Impregnation chamber (a product of Allied High Tech Products, Inc.). The vacuum forced the entrapped air out from within sample and caused the epoxy to fill the voids, cracks or open spaces in matrix. Epoxy impregnated specimen was cured at room temperature as per instructions given by Epoxy resin manufacturer.

3. Preparation of polished specimens

Once the epoxy resin was set, specimen was subjected to polishing. MD-System by Struers A/S was used for grinding and polishing of specimens. MD-disc, MD-Piano, MD-Dac, MD-Chem discs together form the MD-System. MD-Disc served as the magnetic supporting disc for the MD-Piano series, MD-Dac and MD-Chem discs.

Process of polishing involved two distinct stages, i.e. grinding and polishing. Grinding aims towards removal of initial surface defects. MD-Piano grades 80, 220, 600, 1200 producing a scratch pattern comparable to silicon carbide paper of corresponding grit sizes were used for grinding. Mineral oil was used as lubricant during the grinding process. Polishing aims towards a scratch free, mirror finished surface. Struers MD-DAC

and Struers MD-Chem polishing cloths were used for polishing. Propylene glycol was used as lubricant during the polishing process. Specimen was cleaned with Isopropanol after every grinding/polishing cycle to remove grit.

4.6 X-Ray Diffraction

Mortar specimens using Type I Low alkali cement, reactive aggregate subjected to 38°C and solutions like de-ionized water, 3% Sodium chloride, 3% Calcium chloride and 3% Potassium chloride solution were used for this test. Test samples were prepared by crushing mortar specimens to a fine form. The test was executed using Rigaku Ultima IV multipurpose X-ray diffraction system in Chemistry Department, Clemson University. Figure shows X-ray diffraction patterns for compounds of interest.

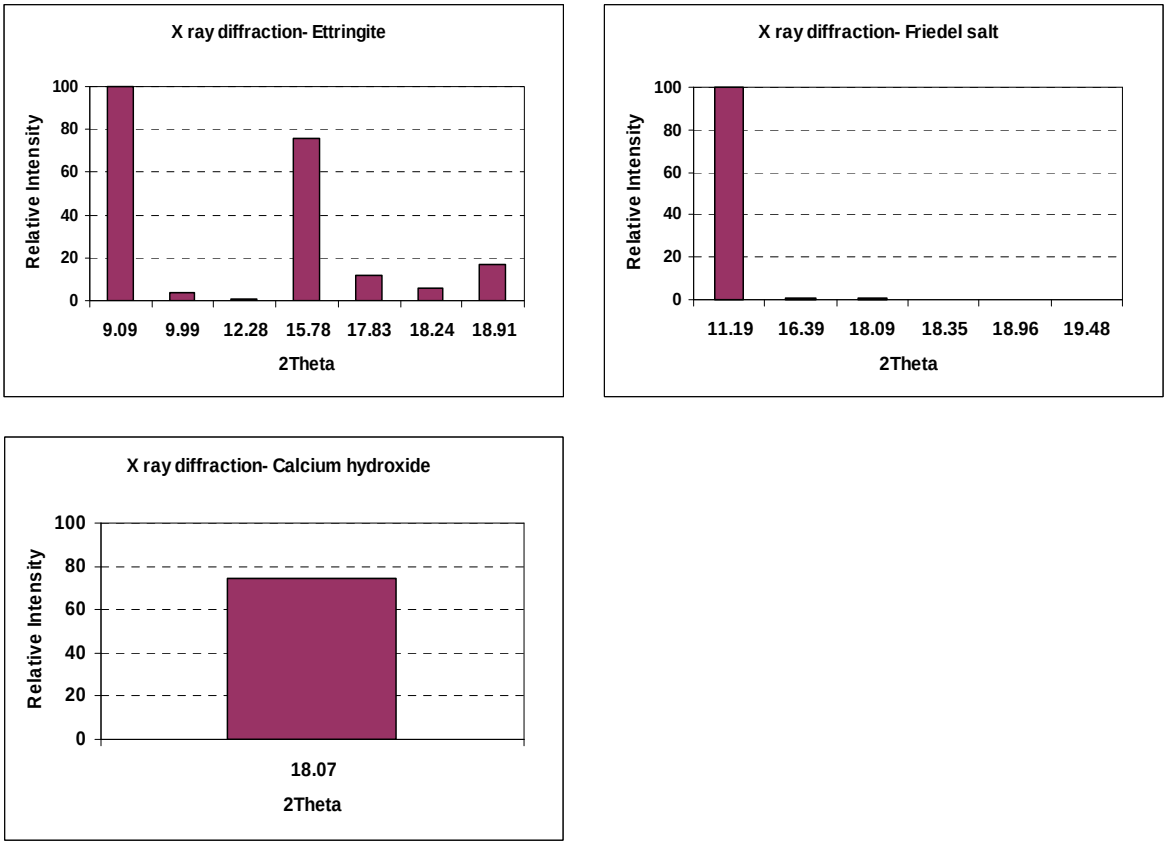


Figure 4.1: X-ray Diffraction Patterns for Compounds of Interest

4.7 Experimental Program

4.7.1 Test Matrix- Standard ASTM C 1260 and Mortar Bar Test

Table below shows the test matrix for Standard ASTM C 1260 and Mortar bar test including variables like aggregate, cement, temperature and deicer solution.

Table 4.1: Test matrix for Standard ASTM C 1260, Mortar Bar test

Test	Soak Solution	Temperature		Cement type		Aggregate type	
		38°C	80°C	Type I High	Type I Low	Sand	FS +Sand
Mortar bar test	De-ionized water	x	x	x	x	x	x
Mortar bar test	3% NaCl	x	x	x	x	x	x
Mortar bar test	3% $CaCl_2$	x	x	x	x	x	x
Mortar bar test	3% KCl	x	x	x	x	x	x
ASTM C 1260	1N NaOH		x	x	x	x	x
Mortar bar test	1N NaOH	x		x	x	x	x
Mortar bar test	0.5N NaOH+ 3% NaCl	x	x	x	x	x	x
Mortar bar test	3% $MgCl_2$	x	x	x	x	x	x

4.7.2 Test Matrix- SEM/EDX analysis

Table below shows the test matrix for SEM/EDX analysis including variables like aggregate, cement, temperature and deicer solution.

Table 4.2: Test matrix for SEM/EDX analysis

Soak Solution	Temperature		Cement type		Aggregate type
	38°C	80°C	Type I High	Type I Low	FS+ Sand
De-ionized water	x	x	x	x	x
3% NaCl	x	x	x	x	x
3% $CaCl_2$	x	x	x	x	x
3% KCl	x	x	x	x	x
1N NaOH	x	x	x	x	x
0.5N NaOH+ 3% NaCl	x	x	x	x	x
3% $MgCl_2$	x	x	x	x	x

4.7.3 Test Matrix- Titrations, ICP Test

Table below shows the test matrix for Titrations and ICP test.

Table 4.3: Test matrix for Titrations, ICP Test

Soak Solution	Temperature	Cement type	Aggregate type
	Room temperature	Type I Low	FS+ Sand
De-ionized water	x	x	x
3% NaCl	x	x	x
3% $CaCl_2$	x	x	x
3% KCl	x	x	x
1N NaOH	x	x	x
0.5N NaOH+ 3% NaCl	x	x	x
3% $MgCl_2$	x	x	x

CHAPTER FIVE

RESULTS AND DISCUSSION

5.1 General

This chapter discusses the results of various tests carried out to predict the effect of chloride deicer. Further, it also looks into the results obtained from SEM/EDX analysis, titrations, ICP test and X-ray diffraction. Results were analyzed to understand the reaction mechanism at micro-structure level under the effect of external soak solutions used in the research.

5.2 Preliminary Investigation of Alkali silica reaction

Fractured samples of mortar bars (subjected to sodium chloride, potassium chloride, 1N Sodium hydroxide solution at 80°C) showed signs of ASR gel when viewed under the optical microscope. There was no gel visible in specimens subjected to de-ionized water at 80°C.

5.3 Results from Standard ASTM C 1260 test

Expansion (%) results for mortar specimens using reactive aggregate- fused silica were compared with expansion (%) results for mortar specimens using non-reactive aggregate- natural sand (Control). Figure (5.1) shows the expansion results of control mortar bars and mortar bars involving fused silica. Mortar bars with fused silica expanded more than mortar bars with natural sand. Mortar specimens using fused silica expanded by about 1.6-1.7 % when stored in 1N NaOH solution at 38°C for 161 days. The expansion results for similar specimens stored in soak solution of same concentration at 80°C for a period of 28 days was found to be around 1.5%. Performance trend of

expansion in mortar bars using fused silica indicate that ASR under the influence of 1N NaOH solution is unaffected by the type of cement. Also, at a given age of 28 days, mortar bars at 80°C expanded more than those at 38°C. Expansion results were far beyond the expansion limit of 0.1% (at 14 days) specified in the test method. This confirms the potential of fused silica to cause alkali silica reaction in the presence of external alkalis.

5.4 Results from Mortar Bar test

This section illustrates the expansion results for mortar specimens in the presence of external alkalis. Expansion results for mortar specimens using non-reactive aggregate-natural sand serve as the Control test results and are compared with performance of mortar specimens using reactive aggregate- fused silica. Results discussed will be in context of mortar bars using fused silica as aggregate.

5.4.1 De-ionized water as soak solution

Figure (5.2) shows the performance trend of mortar bars in terms of expansion (%) versus age of exposure. Results show that mortar specimens using high alkali cement expanded more than those using low alkali cement. It seems clear from the results that alkalis in high alkali cement have the potential (expansion greater than 0.1% at 14 days in Mortar Bar test) to cause alkali silica reaction in concrete at lower temperatures. At 28 days, mortar specimens at 80°C expanded less than those at 38°C. Mortar specimens using fused silica showed a dramatic increase in expansion within 7 days after which expansion slowed down. This may suggest that alkalis are no longer available for the reaction after 7 days.

5.4.2 3% Sodium chloride as soak solution

Expansion (%) results of mortar specimens subjected to 3% sodium chloride soak solution are shown in Figure (5.3). Mortar bars immersed in 3% sodium chloride solution have expanded far beyond the 0.1 % limit. This clearly reflects the potential of sodium chloride to cause alkali silica reaction in concrete. There is a constant increase in expansion with age for all mortar specimens using fused silica at 38°C. This indicates that enough alkalis and hydroxyl ions are available at reaction sites to progress the reaction at a constant rate. At 38°C, maximum recorded expansion was 2.21% at 147 days age. At 80°C, maximum recorded expansion was 1.6-2.0 % at 28 days. This is higher than the expansion observed in mortar bars subjected to 38°C at 28 days. Performance of mortar bars using Type I low alkali cement and Type I high alkali cement are comparable.

5.4.3 3% Calcium chloride as soak solution

Figure (5.4) shows results for expansion in mortar specimens exposed to 3% calcium chloride soak solution. These results are comparable to expansion in mortar specimens exposed to de-ionized water. It seems that expansion effect is caused by alkalis present in the cement. Calcium chloride does not seem to cause alkali silica reaction in mortar specimens at 38°C and 80°C. Mortar bars at 80°C expanded less than mortar bars at 38°C

5.4.4 3% Potassium chloride as soak solution

Figure (5.5) shows the performance trend of mortar specimens, subjected to 3% Potassium chloride solution, in terms of expansion (%) versus age of exposure.

Percentage expansion was recorded as about 2.70% (at 161 days) in mortar specimens at 38°C using fused silica. At 80°C the expansion in mortar bars was found to be 1.07% (at 28 days). Type of cement does not seem to influence the expansion results of mortar bars. Higher expansion results are observed at higher temperature. Expansion greater than 0.1% at 14 days reflects the potential of potassium chloride to cause alkali silica reaction in concrete. At a given age, potassium chloride exposed mortar bars seem to be expanding more than sodium chloride exposed specimens when subjected to a lower temperature (here, 38°C). Rate of expansion in mortar bars seems to be constant. This implies a constant supply/availability of alkalis and hydroxyl ions at reaction sites.

5.4.5 0.5N Sodium hydroxide and 3% Sodium chloride blended soak solution

Figure (5.6) shows the expansion of mortar specimens subjected to above soak solution under different conditions. At 38°C expansion in mortar bars was about 2.45% (at 161 days). At 80°C mortar specimens expanded by about 1.5% (at 28 days). Expansion in mortar bars was found to be more than expansion in mortar bars subjected to 1N Sodium hydroxide solution. This implies that Cl^- ions play an important role in the alkali silica reaction process. Expansion results for specimens cast with Type I Low and High alkali cement are comparable. Higher temperature seems to accelerate the expansion reaction.

5.4.6 3% Magnesium Chloride as soak solution

Figure (5.6) shows the expansion of mortar specimens subject to 3% Magnesium chloride soak solution under different conditions. Expansion results are comparable to

expansion in mortar specimens subjected to de-ionized water. This suggests that magnesium chloride does not seem to have any effect on alkali silica reaction in concrete. The expansive effect is due to alkalis in cement.

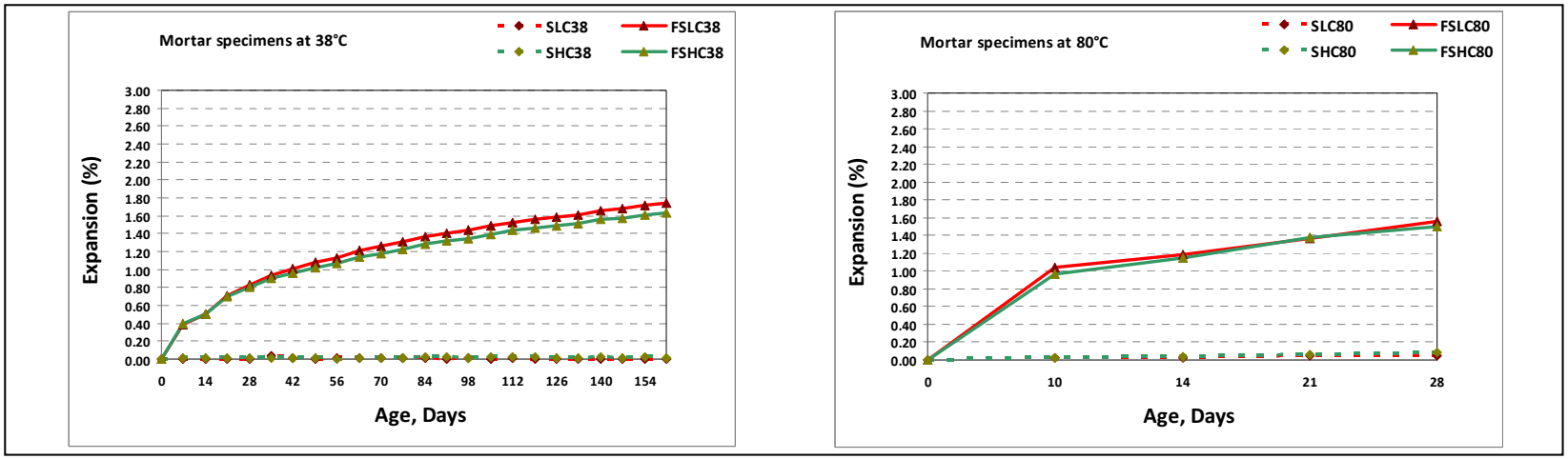


Figure 5.1: Expansion results of Mortar bars in Standard ASTM C 1260 subjected to different temperature, cement and aggregate as per test matrix

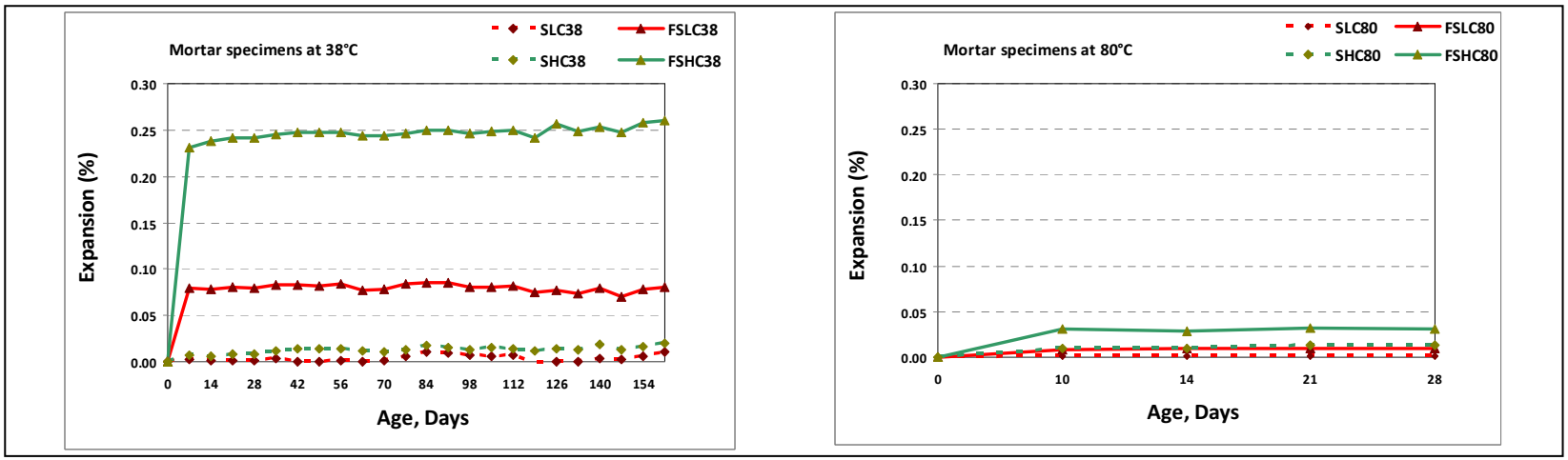


Figure 5.2: Expansion results of Mortar bars in De-ionized water subjected to different temperature, cement and aggregate as per test matrix

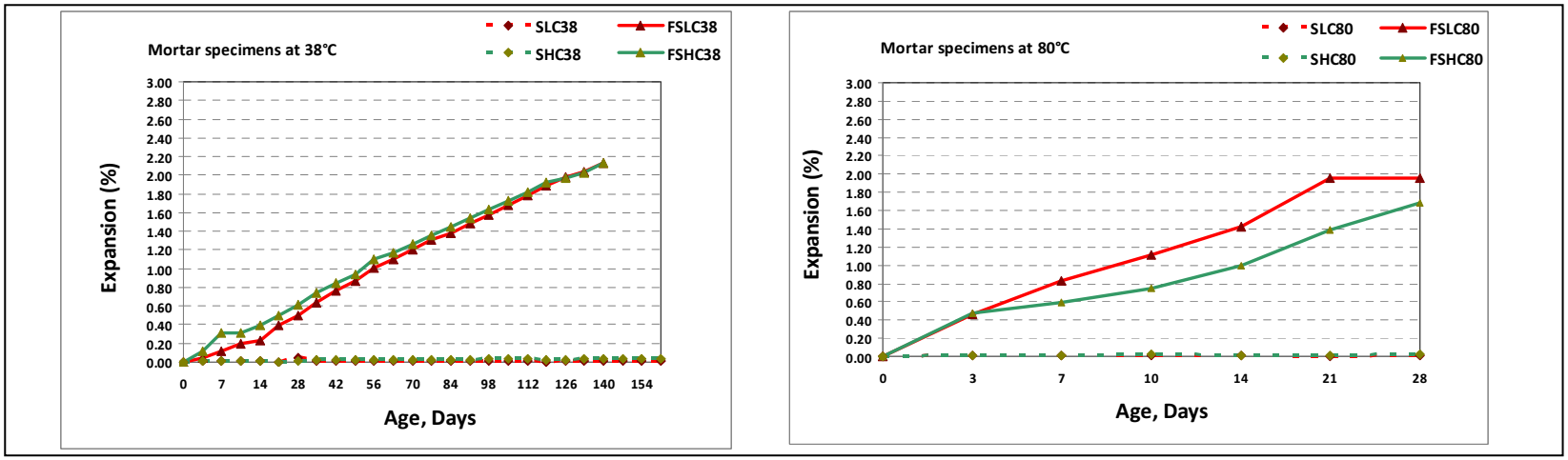


Figure 5.3: Expansion results of Mortar bars in 3% Sodium chloride solution subjected to different temperature, cement and aggregate as per test matrix

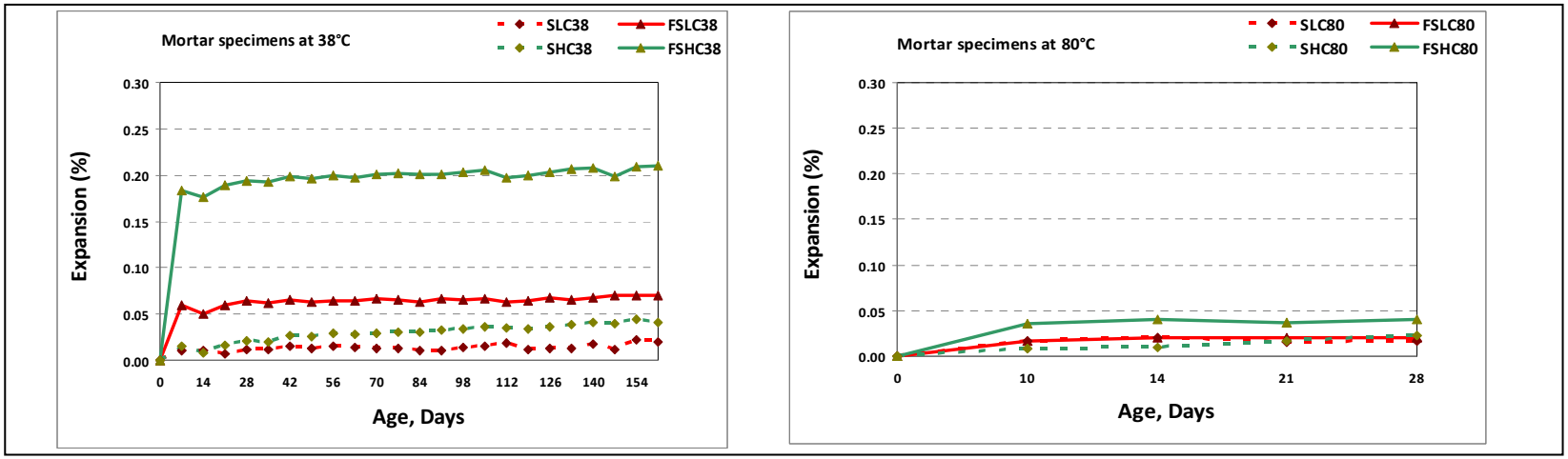


Figure 5.4: Expansion results of Mortar bars in 3% Calcium chloride solution subjected to different temperature, cement and aggregate as per test matrix

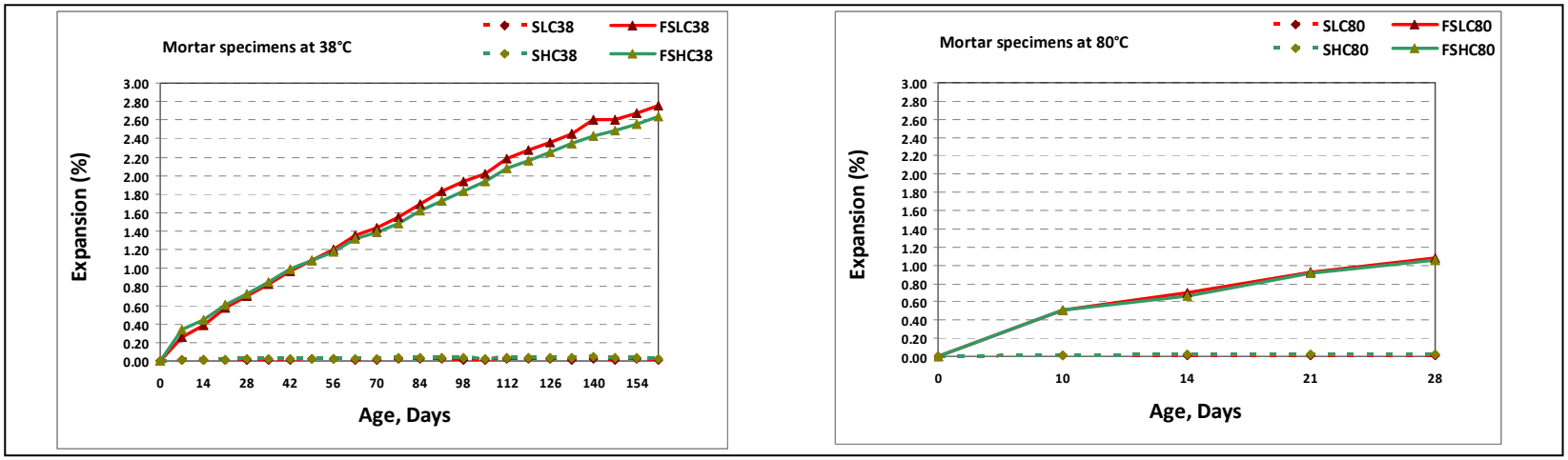


Figure 5.5: Expansion results of Mortar bars in 3% Potassium chloride solution subjected to different temperature, cement and aggregate as per test matrix

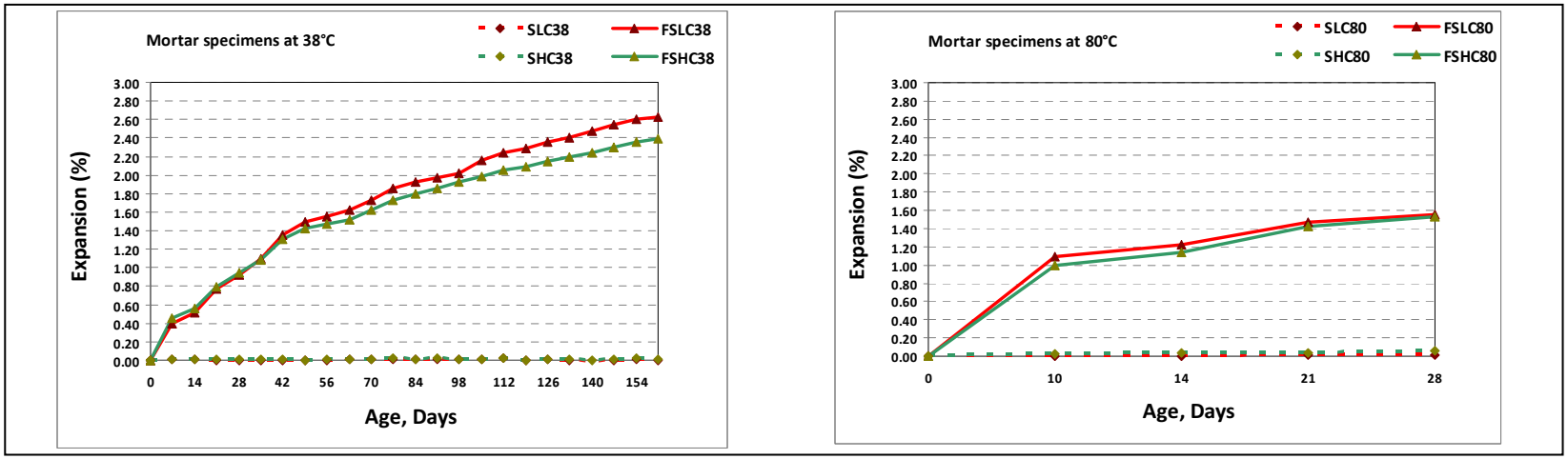


Figure 5.6: Expansion results of Mortar bars in 0.5N Sodium hydroxide and 3% Sodium chloride blended solution subjected to different temperature, cement and aggregate as per test matrix

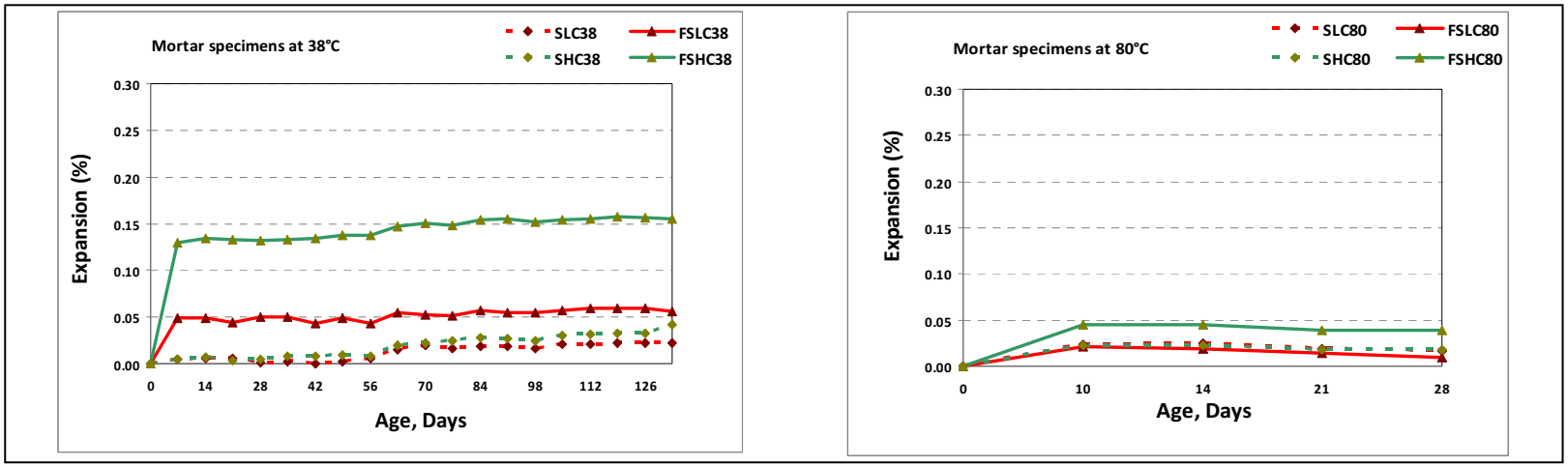


Figure 5.7: Expansion results of Mortar bars in 3% Magnesium chloride solution subjected to different temperature, cement and aggregate as per test matrix

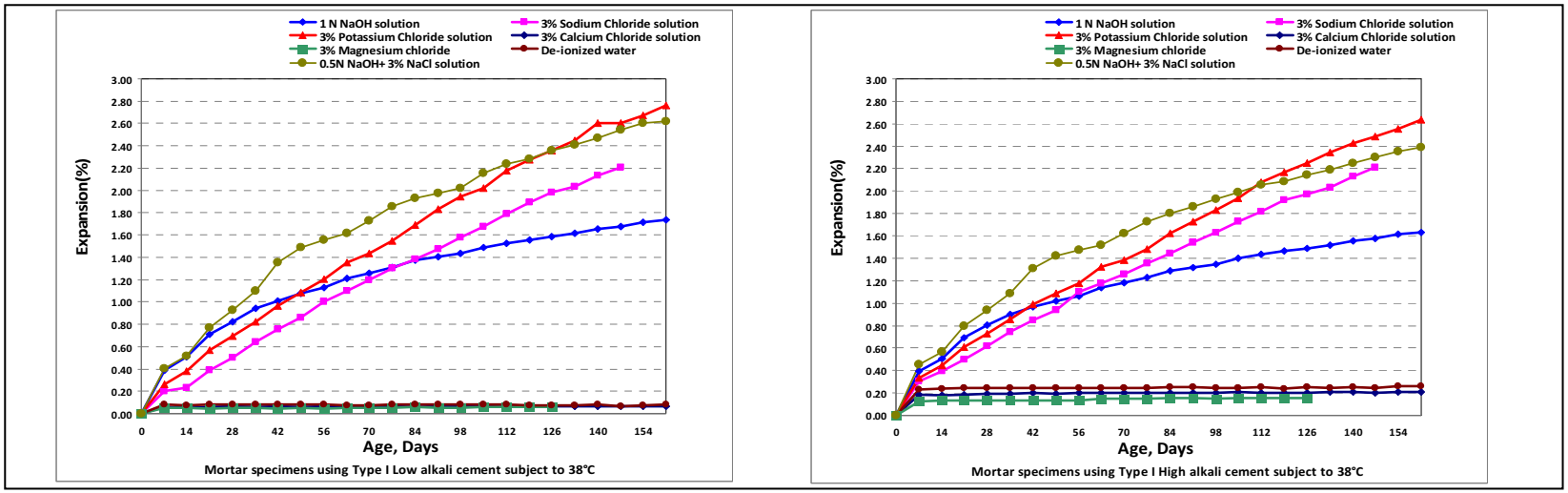


Figure 5.8: Influence of alkali content of cement, alkali type on Expansion of (Fused silica+ Sand) mortar bars

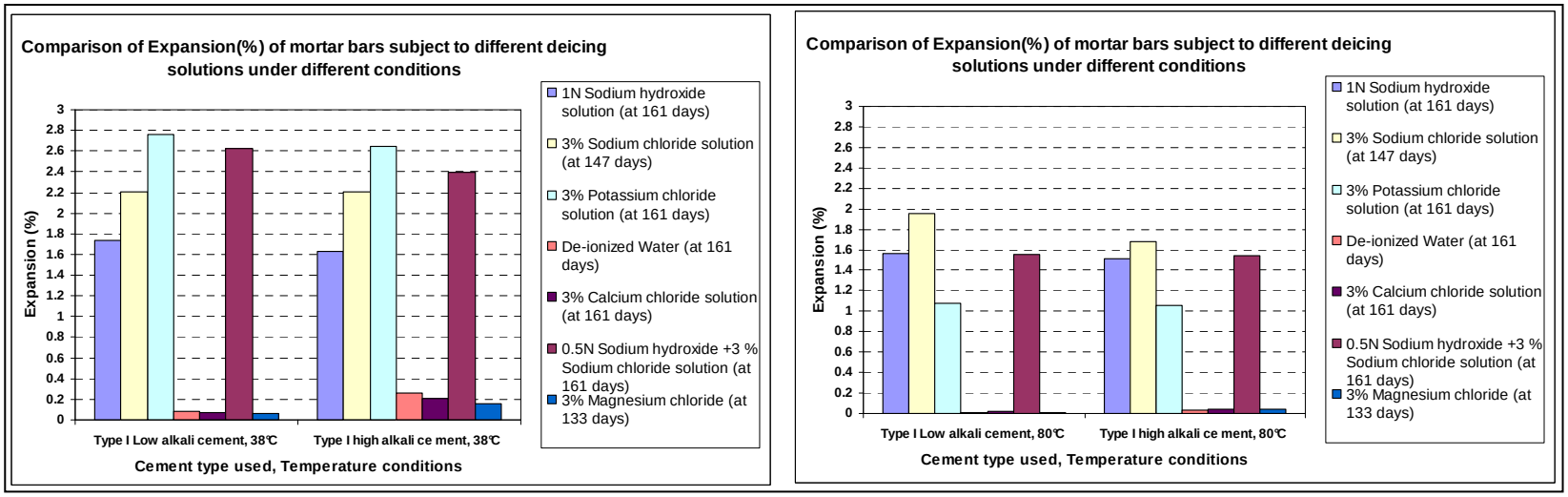


Figure 5.9: Influence of Storage temperature on expansion of (Fused silica+ Sand) mortar bars

5.6 Results from Titrations

Hydroxyl ion concentration is an important requirement for an alkali silica reactive environment. Crushed mortar specimens were subjected to different soak solutions as per the test matrix for a period of about 28 days at room temperature. Filtered solution was diluted and used for titration experiments to find the hydroxyl ion concentration in the water soluble hydroxides. Figure (5.10) shows the hydroxyl ion concentration in water soluble hydroxides. It is observed that hydroxyl ion concentration is very high in solution filtered from crushed mortar specimens subjected to 1N NaOH solution and (0.5N NaOH+ 3% Sodium chloride) solution compared to other solutions. Hydroxyl ion concentration in the solution filtered from crushed mortar specimens subjected to chloride salt solutions and de-ionized water are comparable.

Low hydroxyl ion concentrations in solution filtered from crushed mortar samples subjected to chloride salt solutions suggest the absence of ASR. Although, mortar bar test results show that mortar bars subjected to alkali chloride solutions expand more than those subjected to alkali hydroxide solutions. This implies that it is possible that formation of chloroaluminates released the extra OH^- ions in the pore solution. As a result, we observed ASR effect in the mortar specimens. It also suggests that transformation from ettringite to chloroaluminates is a timely process. These test results are not conclusive and need further investigation.

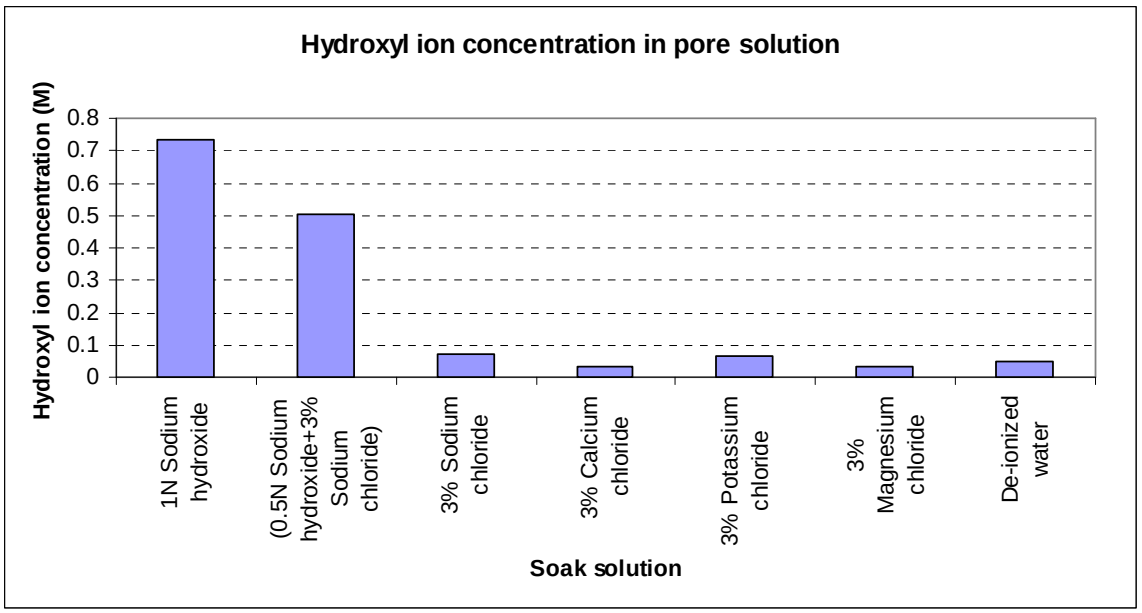


Figure 5.10: OH^- ion concentration in water soluble hydroxides of mortar Specimens ... using Type I Low alkali cement and (Fused silica+ sand) as aggregate subjected to different deicing solutions at 38°C temperature

5.7 Results from X-ray Diffraction

X-ray diffraction was performed on finely powdered mortar specimens with 2-theta value from 3° to 20° and scan speed of 0.16 cps. Based on peaks observed and study of standard X-ray diffraction patterns of compounds of interest, presence of hydration products was predicted. Mortar samples subjected to 3% sodium chloride (Figure 5.12), 3% potassium chloride (Figure 5.14) and 3% calcium chloride (Figure 5.13) showed low intensity distinct peaks of Friedel salt at about 11°. Distinct peaks of ettringite are observed in all samples. Mortar samples subjected to de-ionized water (Figure 5.11) and 3% calcium chloride showed distinct peak of calcium hydroxide at about 18° which suggest absence of ASR. Table (5.1) gives details of peaks observed in each sample.

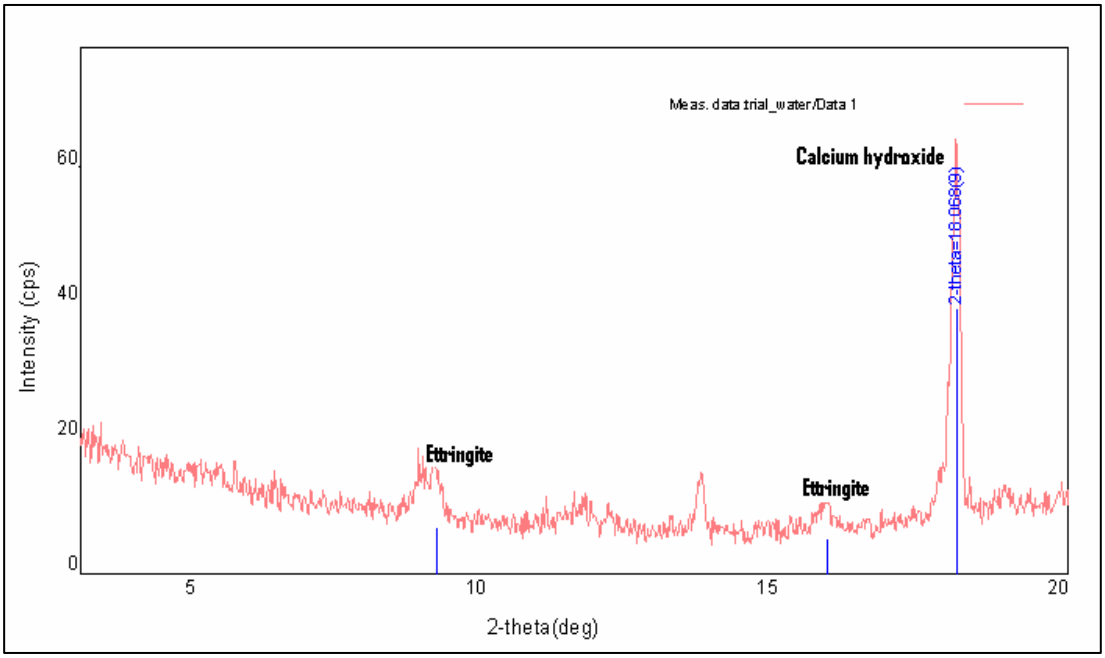


Figure 5.11: X ray diffraction results for Mortar specimens using (Fused silica+ sand) as aggregate, Type I Low alkali Cement subjected to 38°C and De-ionized water

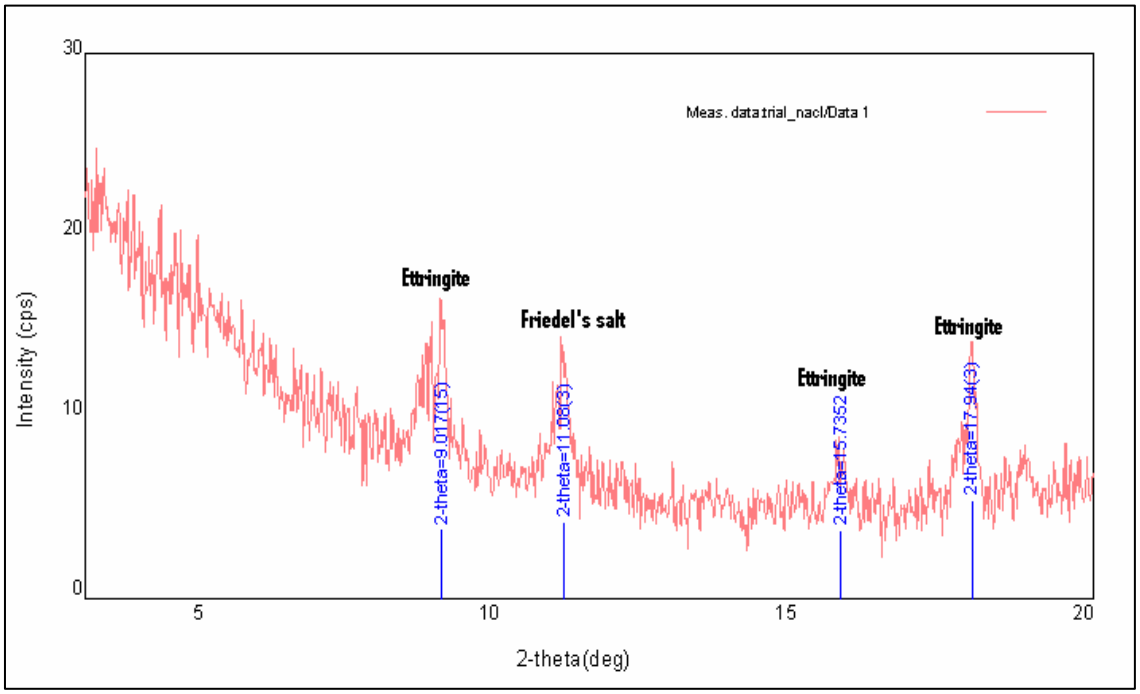


Figure 5.12: X ray diffraction results for Mortar specimens using (Fused silica+ sand) as aggregate, Type I Low alkali Cement subjected to 38°C and 3% Sodium .. chloride solution

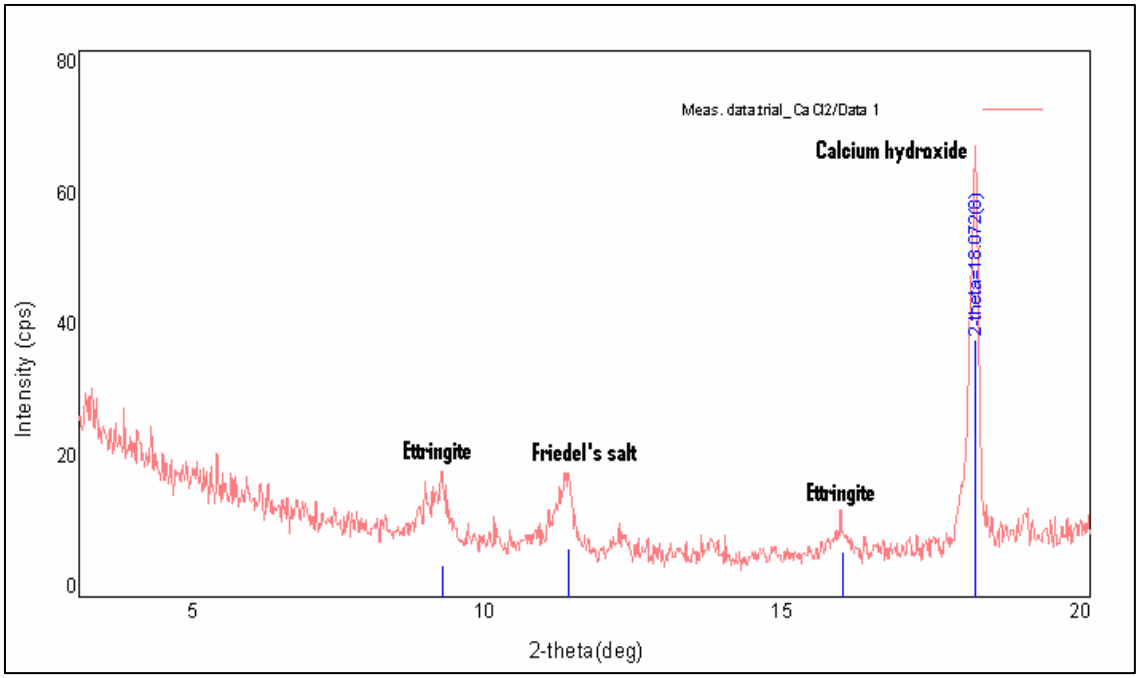


Figure 5.13: X ray diffraction results for Mortar specimens using (Fused silica+ sand) as aggregate, Type I Low alkali Cement subjected to 38°C and 3% Calcium . chloride solution

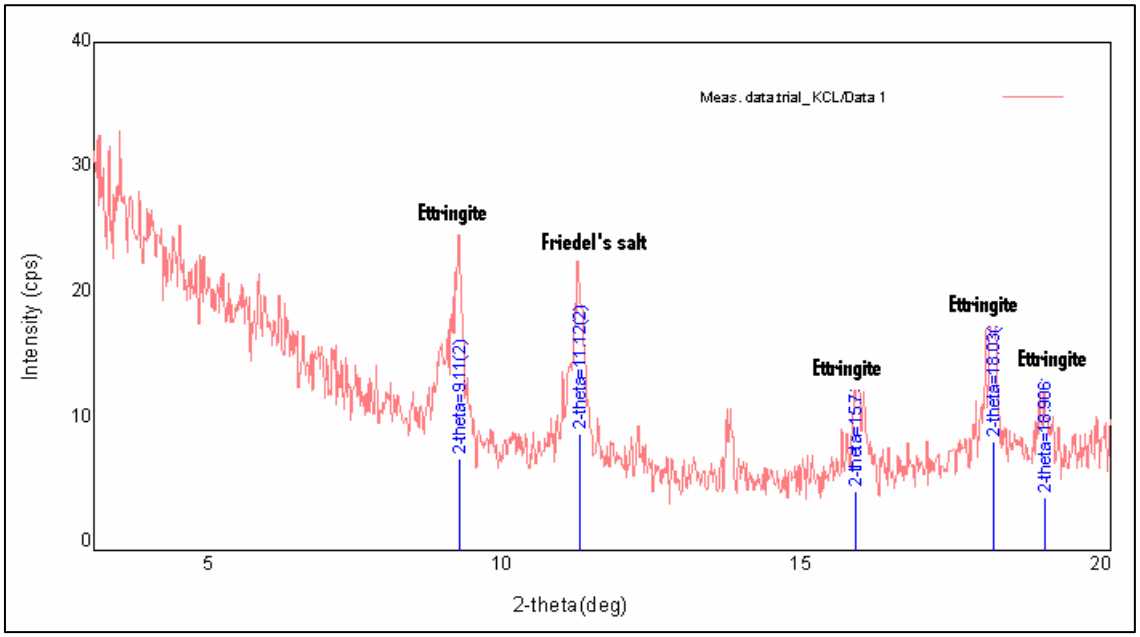


Figure 5.14: X ray diffraction results for Mortar specimens using (Fused silica+ sand) as aggregate, Type I Low alkali Cement subjected to 38°C and 3% Potassium chloride solution

Table 5.1: Details of X-ray diffraction peaks

Sample ID	Soak solution	Peaks observed (2 theta)	Phase name/ Hydration products predicted
0	De-ionized water	9.13	Ettringite
		15.85	Ettringite
		18.068	Calcium hydroxide
1	3% Sodium chloride solution	9.017	Ettringite
		11.08	Friedel's salt
		15.735	Ettringite
		17.94	Ettringite
2	3% Calcium chloride solution	9.12	Ettringite
		11.22	Friedel's salt
		15.824	Ettringite
		18.072	Calcium hydroxide
3	3% Potassium chloride solution	9.11	Ettringite
		11.12	Friedel's salt
		15.73	Ettringite
		18.03	Ettringite
		18.906	Ettringite

5.7 Results from Visual, SEM/ EDX analysis

Figure (5.15) to Figure (5.21) are Scanning electron micrograph and X-ray elemental spectra of ASR gel in air void, ASR gel around aggregate and mortar matrix. The specimens used for this study were as per the test matrix for SEM/ EDX analysis (Section 4.7.2).

1. Mortar specimens subjected to de-ionized water

Mortar samples showed no signs of cracks. Figure (5.15) shows SEM micrograph and EDX spectra for mortar matrix. SEM study reveals no signs of alkali silica reaction. Results from EDX spectra show that for samples involving different

cement and exposed to different temperature, the alkali/ silica ratio is comparable. Alkali/ silica ratio in different samples with variation in cement type and temperature appears to be around 0.05.

2. Mortar specimens subjected to 3% Sodium chloride solution

Samples exposed to 38°C show visible continuous hair crack along the length of the specimen. In case of samples subjected to 80°C, patterned cracks are observed with major continuous crack being along the length of specimen. These cracks seem to be wider and more intense than cracks developed in mortar bars at lower temperature.

Figure (5.16) shows the Scanning electron micrographs and EDX spectra for mortar samples subjected to 3% Sodium chloride solution. SEM micrographs show the presence of gel in cracked aggregate and in air voids in all specimens irrespective of exposure temperature or type of cement used. EDX spectra of this gel show an alkali/ silica ratio of 0.1-0.4 (> 0.05, i.e. alkali/ silica ratio observed in mortar bars subjected to de-ionized water). This confirms that the gel is ASR gel. EDX spectra results of mortar matrix show alkali/ silica ratio greater than 0.05. Thus, it is likely that ASR gel had migrated into the matrix where it became rich in calcium due to interaction with cement paste (High Ca/ Si ratio in the paste). Alkalis migrate to other potential reaction sites through the matrix. Micro-cracks found in the mortar matrix may be due to ASR or shrinkage. The X-ray elemental spectra associated with the mortar matrix reflects the presence of chlorides. It is possible that chlorides from the chloride salts have interacted with hydration products to form chloroaluminates.

3. Mortar specimens subjected to 3% Calcium chloride solution

Visual inspection and SEM/ EDX analysis show no signs of ASR. Figure (5.17) reflects SEM micrograph and EDX spectra for matrix. X-ray elemental spectra results for mortar matrix of specimens studied indicate the presence of chlorides in the matrix. Thus, it is likely that chloroaluminates have formed and so hydroxyl ions are available for the reaction. The mix is rich in calcium, but not alkalis. Bringing all the facts together, it seems that excess of calcium in the matrix has reduced ASR potential. This is in agreement with many published literature which explains the formation of non-swelling calcium silicate gel [9,13].

4. Mortar specimens subjected to 3% Potassium chloride solution

Cracks developed in mortar specimens subjected to this solution were more intense and wider than cracks developed in specimens exposed to sodium chloride solution.

Figure (5.18) shows the SEM micrograph and spectra results for mortar specimens subjected to potassium chloride solution with varied temperature and cement. Alkali silica gel is observed in cracked aggregates and also in air voids. The alkali/ silica ratio varies from 0.2-0.6. EDX spectra results reveal the presence of alkalis and chloride ions in the matrix. This suggests the presence of ASR gel and chloroaluminates in the system. Matrix shows lot of micro-cracks which are probably associated with ASR or shrinkage.

5. Mortar specimens subjected to 1N Sodium hydroxide solution

Visual inspection results of mortar bars show intense mapped cracking. Cracks are wider and intense than specimens subjected to sodium chloride and potassium chloride solutions.

SEM micrographs reflect ASR gel in aggregate and air void. Alkali/ silica ratio is observed in the range of 0.2-0.4. Figure (5.19b) reflects an extended case of ASR distress in a cracked aggregate where the alkali/ silica ratio is greater than 1.0. Alkali/ silica ratio is comparable to that observed in sodium chloride and potassium chloride exposed mortar bars. Low calcium/ silica gel is observed in the air void. The intensity and extent of reaction can be attributed to the external source of hydroxyl ions. Mortar matrix spectra results reflect a high alkali/ silica gel which indicate the presence of ASR gel. Calcium/ silica ratio is found to vary based on distance from aggregate.

6. Mortar specimens subjected to 0.5N Sodium hydroxide and 3% Sodium chloride blended solution

Patterned cracking is found in mortar specimens subjected to this solution. Mortar specimens at 80°C show more intense cracks in comparison to mortar specimens at 38°C.

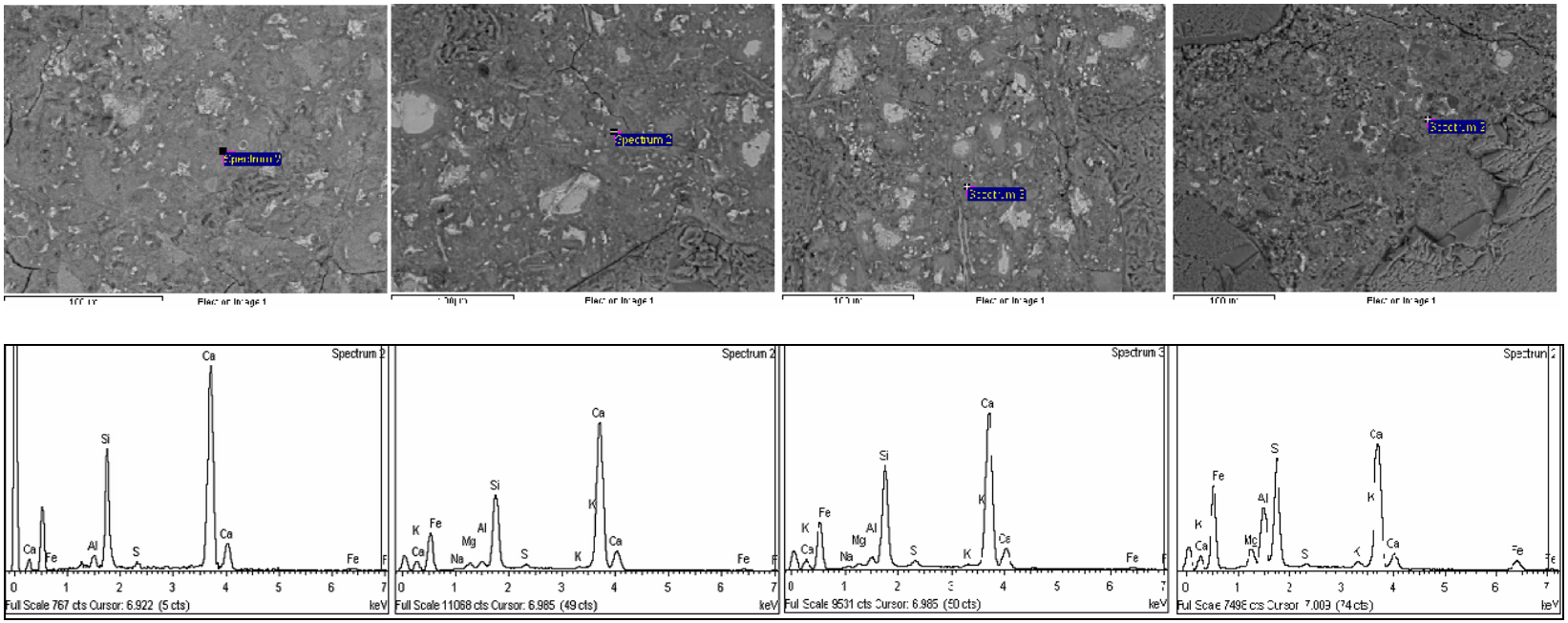
SEM/ EDX analysis results as in Figure (5.20) show presence of alkali silica gel in cracked aggregate, in air void and in matrix. Alkali/ silica ratio is within the range of 0.2-0.5. Calcium/ silica ratio varies based on distance from aggregate.

7. Mortar specimens subjected to 3% Magnesium chloride solution

Figure (5.21) shows SEM/EDX results for mortar specimens using Type I Low/ High alkali cement at 80°C. No signs of ASR are observed. High concentrations of magnesium and chloride ions are found in the matrix.

5.8 Results from ICP Test

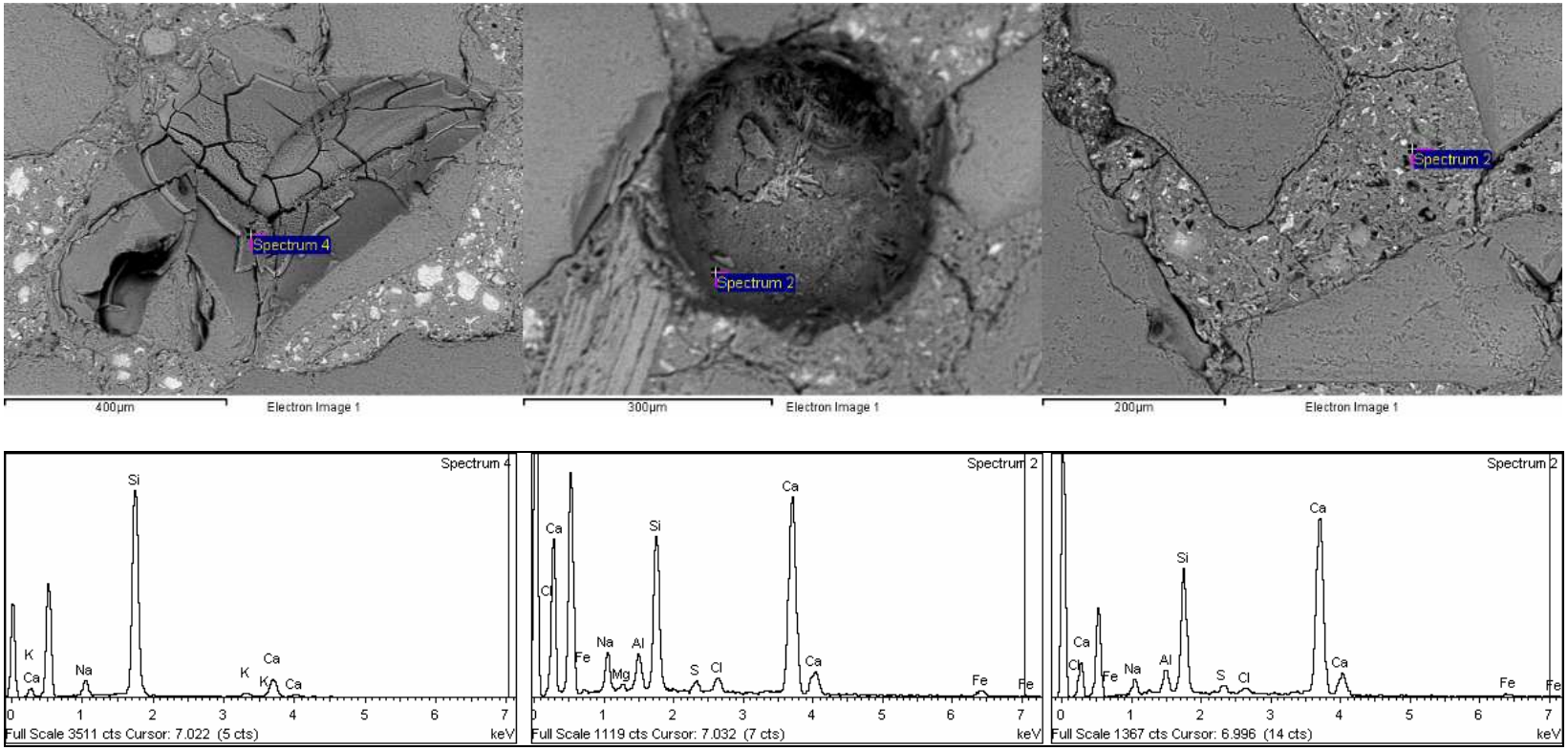
ICP Test results are not conclusive and needs further investigation. Table (A.14) reflects the ICP test results.



a) Alkali/ Silica= - b) Alkali/ Silica= 0.03 c) Alkali/ Silica= 0.02 d) Alkali/ Silica= 0.06

Figure 5.15: SEM/ EDX analysis results: Mortar specimens subjected to De-ionized water

a) Type I Low alkali cement, 38°C
b) Type I High alkali cement, 38°C
c) Type I Low alkali cement, 80°C
d) Type I High alkali cement, 80°C

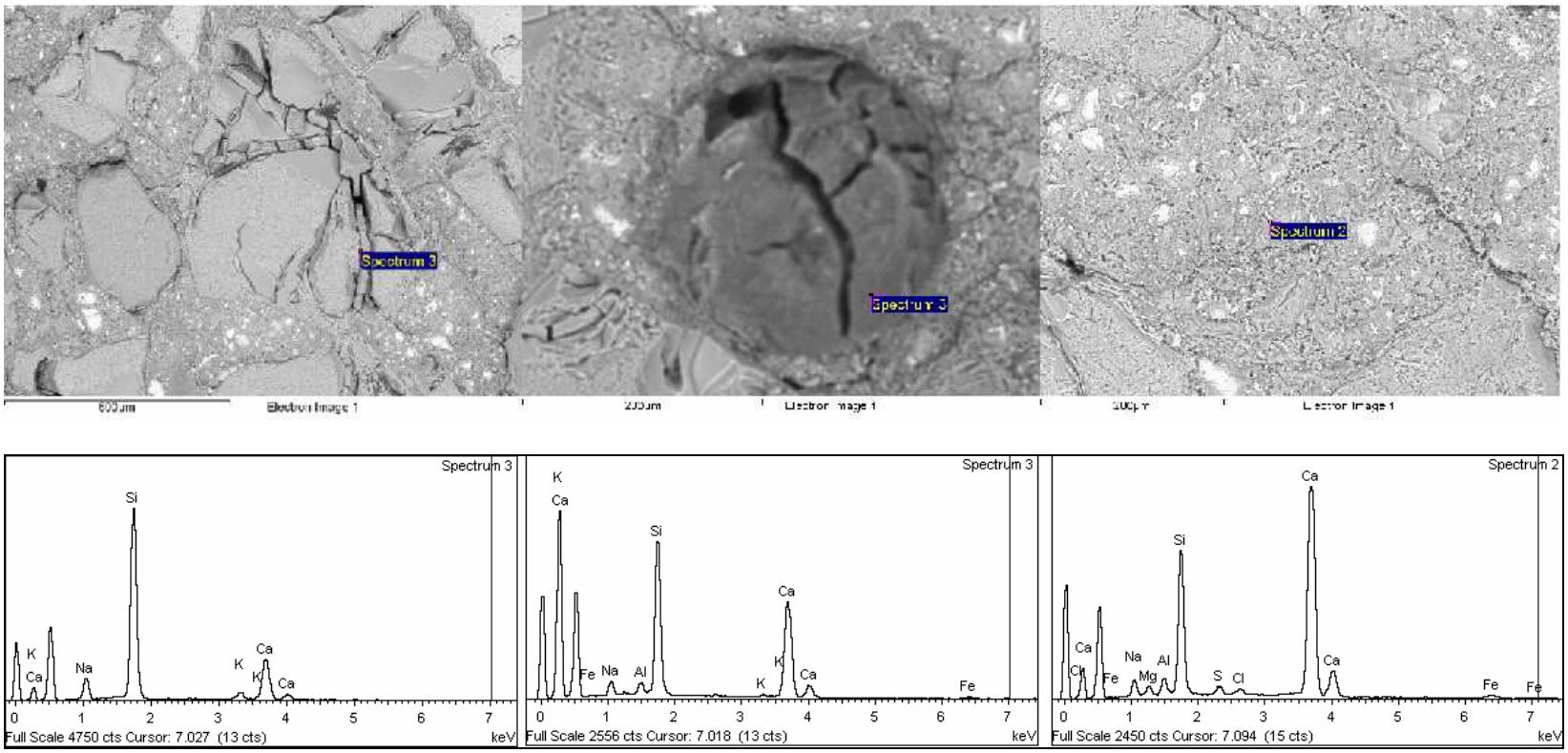


Alkali/ Silica = 0.13
Calcium/ Silica = 0.19

Alkali/ Silica = 0.37
Calcium/ Silica = 2.04

Alkali/ Silica = 0.21
Calcium/ Silica = 2.38
% Chloride = 1.66

Figure 5.16: SEM/ EDX analysis results: Mortar specimens subjected to 3% Sodium chloride solution
a) Type I Low alkali cement, 38°C

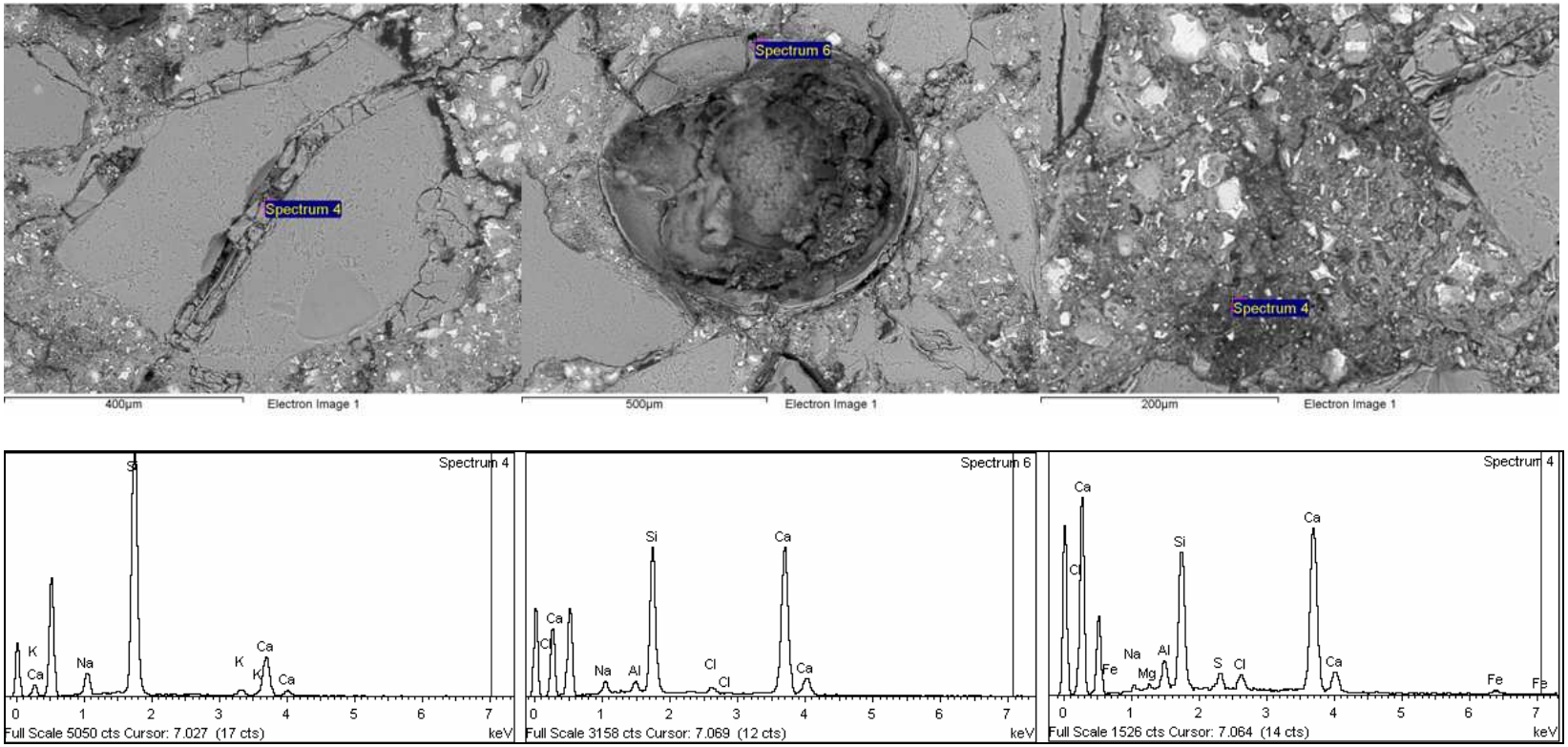


Alkali/ Silica = 0.21
Calcium/ Silica = 0.45

Alkali/ Silica = 0.15
Calcium/ Silica = 1.09

Alkali/ Silica = 0.17
Calcium/ Silica = 2.44
% Chloride = 1.29

Figure 5.16: SEM/ EDX analysis results: Mortar specimens subjected to 3% Sodium chloride solution
b) Type I High alkali cement, 38°C

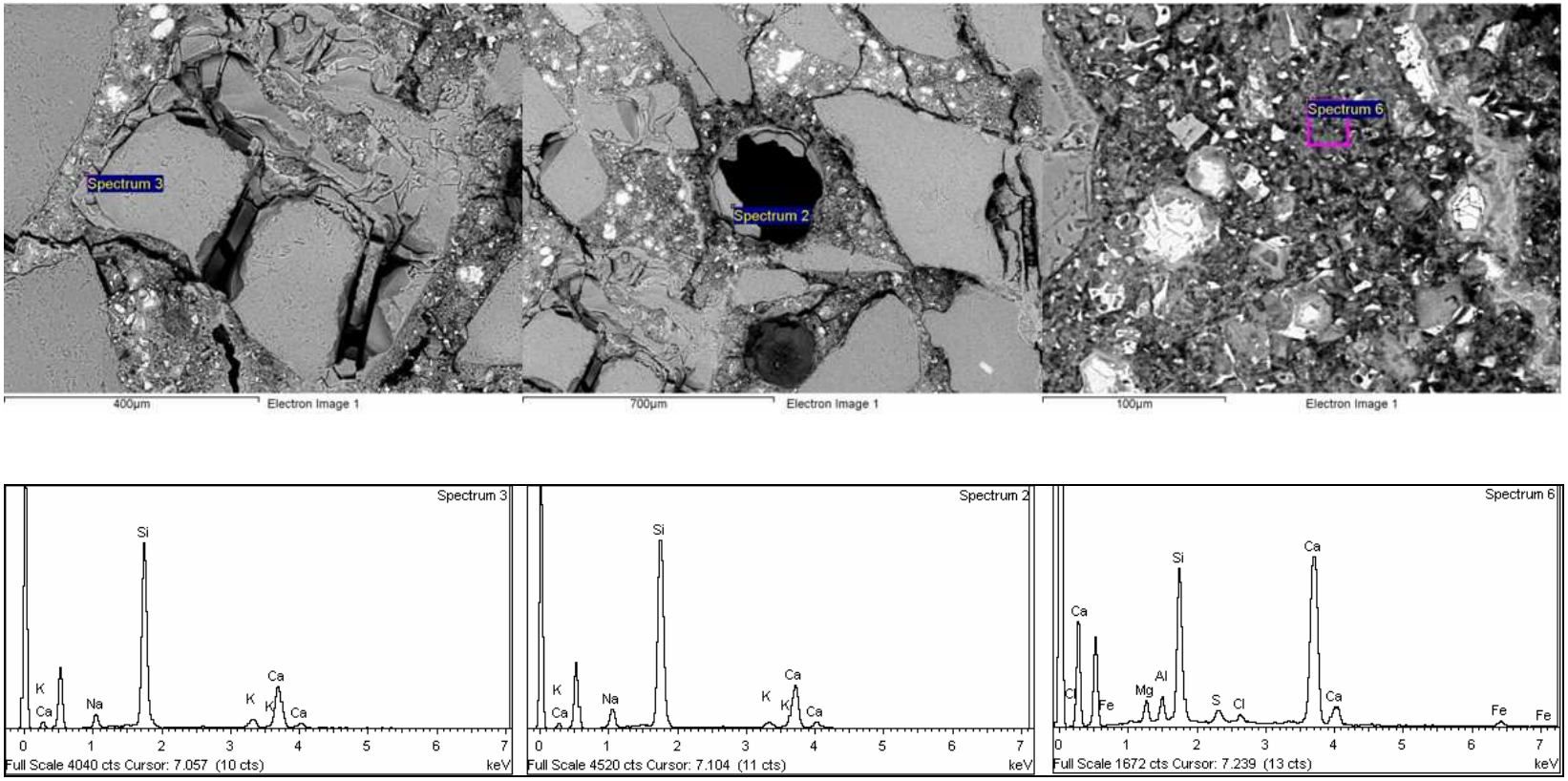


Alkali/ Silica = 0.16
Calcium/ Silica = 0.33

Alkali/ Silica = 0.13
Calcium/ Silica = 1.73

Alkali/ Silica = 0.07
Calcium/ Silica = 2.05
% Chloride = 4.18

Figure 5.16: SEM/ EDX analysis results: Mortar specimens subjected to 3% Sodium chloride solution
c) Type I Low alkali cement, 80°C

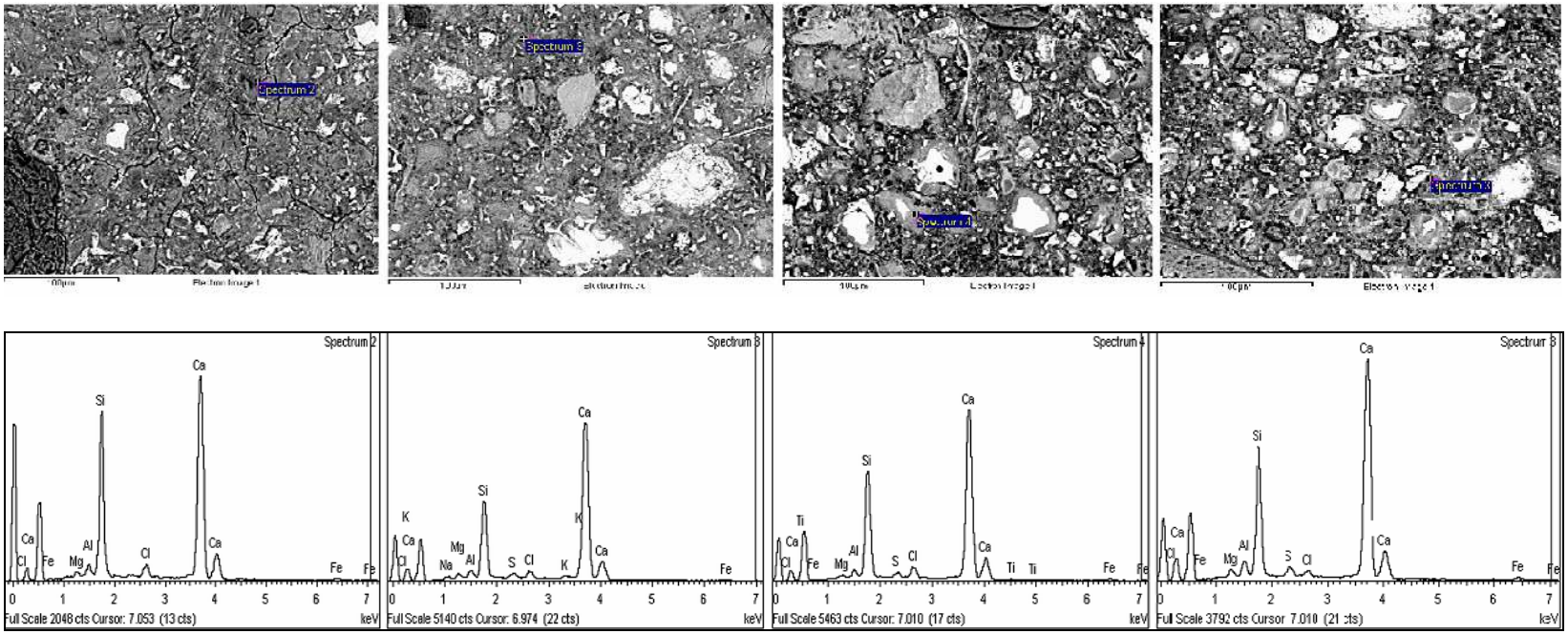


Alkali/ Silica = 0.20
Calcium/ Silica = 0.47

Alkali/ Silica = 0.19
Calcium/ Silica = 0.48

Alkali/ Silica = Nil
Calcium/ Silica = 1.86
% Chloride = 1.91

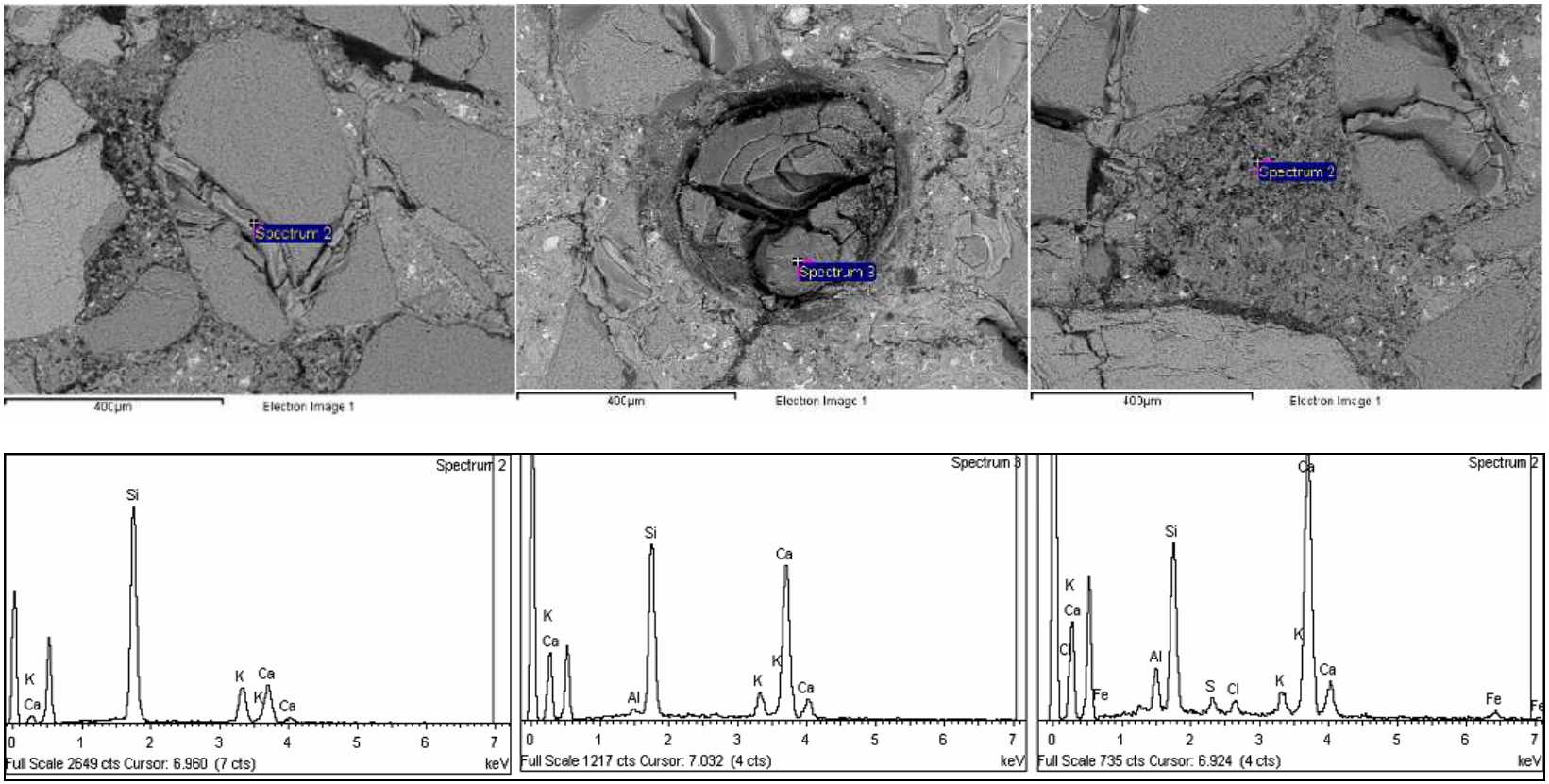
Figure 5.16: SEM/ EDX analysis results: Mortar specimens subjected to 3% Sodium chloride solution
d) Type I High alkali cement, 80°C



a) % Chloride = 3.47 b) % Chloride= 2.49 c) % Chloride= 3.66 d) % Chloride= 1.71

Figure 5.17: SEM/ EDX analysis results: Mortar specimens subjected to 3% Calcium chloride solution

e) Type I Low alkali cement, 38°C
f) Type I High alkali cement, 38°C
g) Type I Low alkali cement, 80°C
h) Type I High alkali cement, 80°C

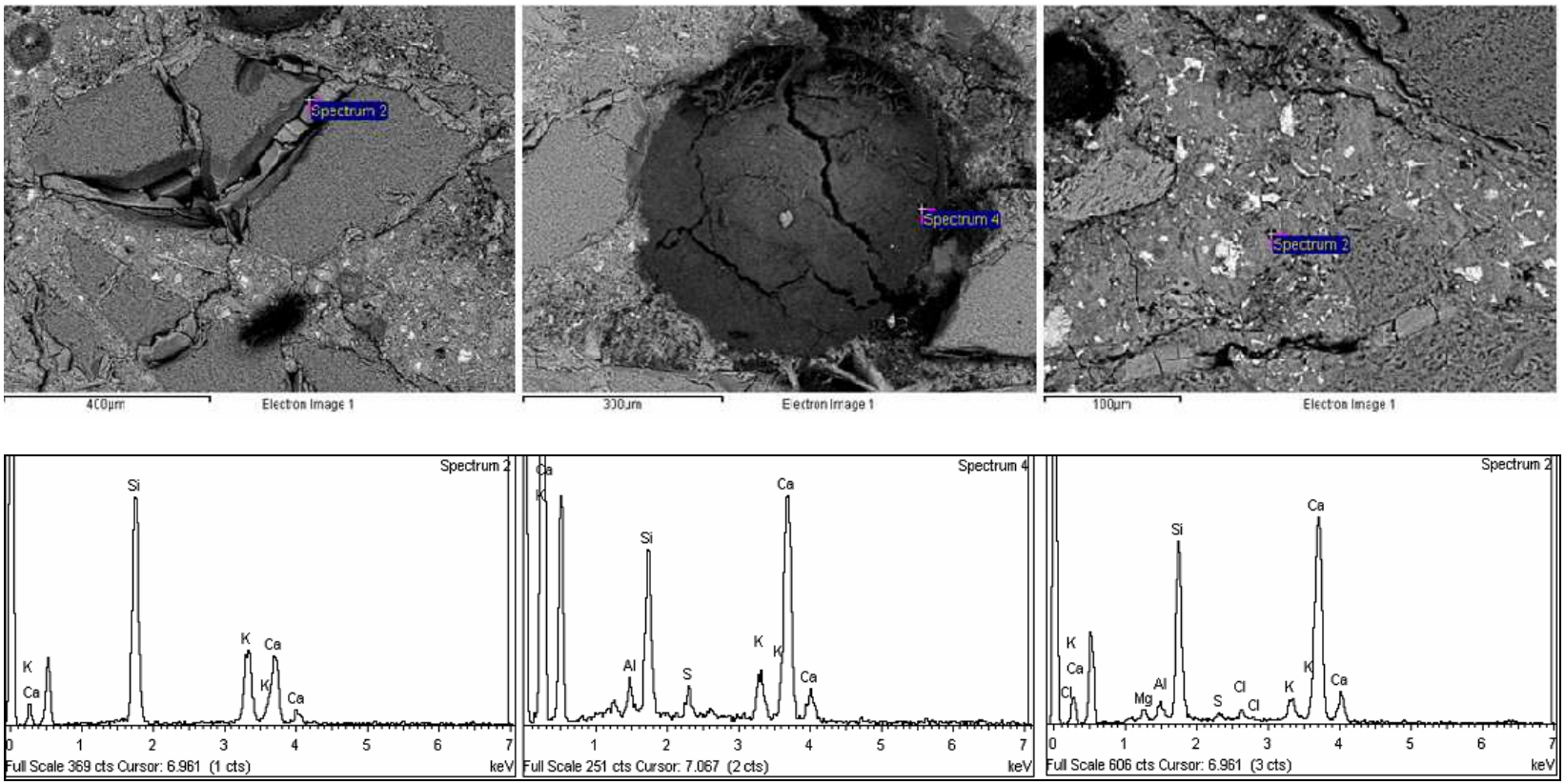


Alkali/ Silica = 0.32
Calcium/ Silica = 0.38

Alkali/ Silica = 0.22
Calcium/ Silica = 1.65

Alkali/ Silica = 0.19
Calcium/ Silica = 2.60
% Chloride = 2.58

Figure 5.18: SEM/ EDX analysis results: Mortar specimens subjected to 3% Potassium chloride solution
a) Type I Low alkali cement, 38°C

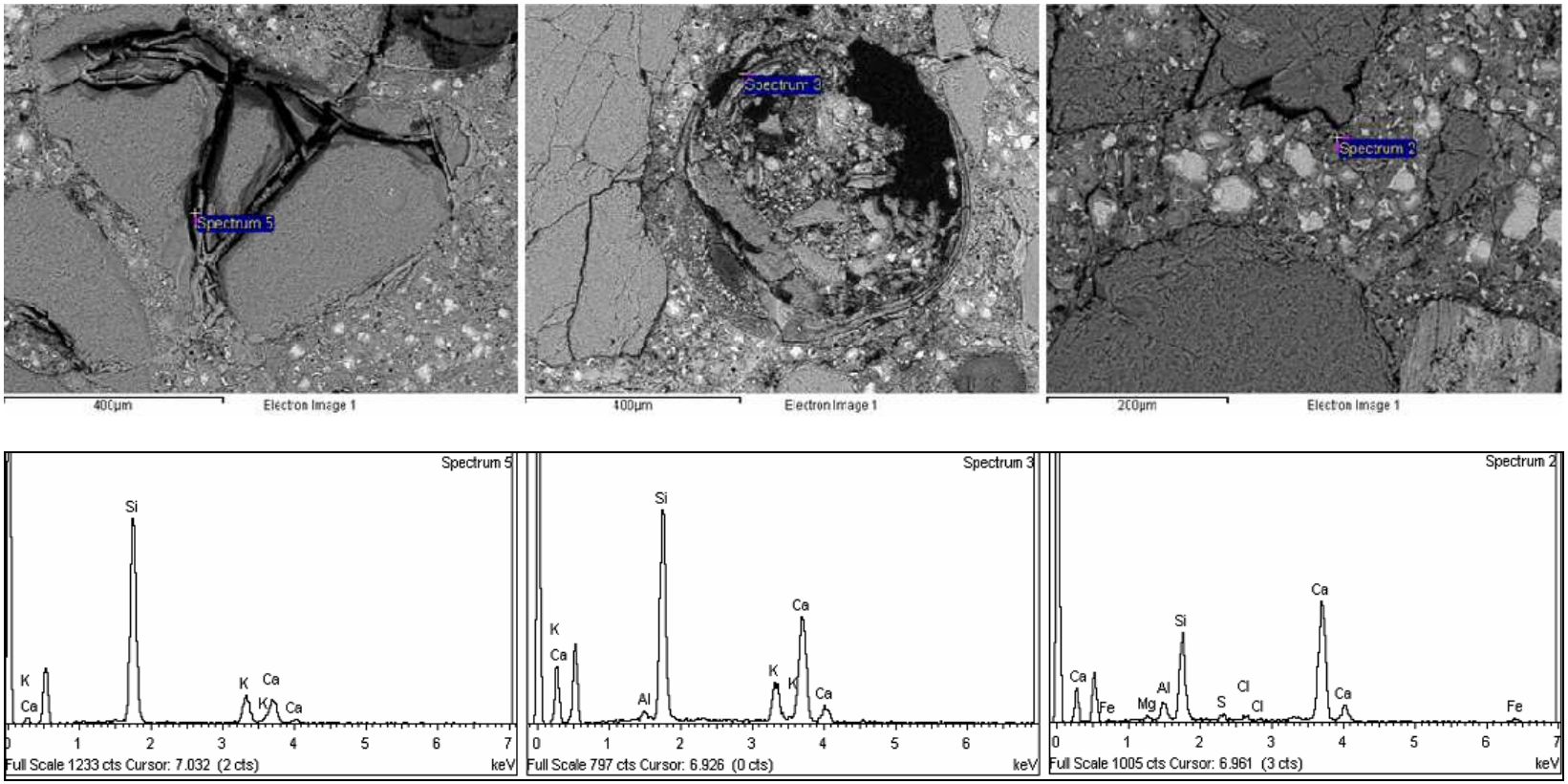


Alkali/ Silica = 0.55
Calcium/ Silica = 0.62

Alkali/ Silica = 0.41
Calcium/ Silica = 2.58

Alkali/ Silica = 0.16
Calcium/ Silica = 2.10
% Chloride = 1.84

Figure 5.18: SEM/ EDX analysis results: Mortar specimens subjected to 3% Potassium chloride solution
b) Type I High alkali cement, 38°C



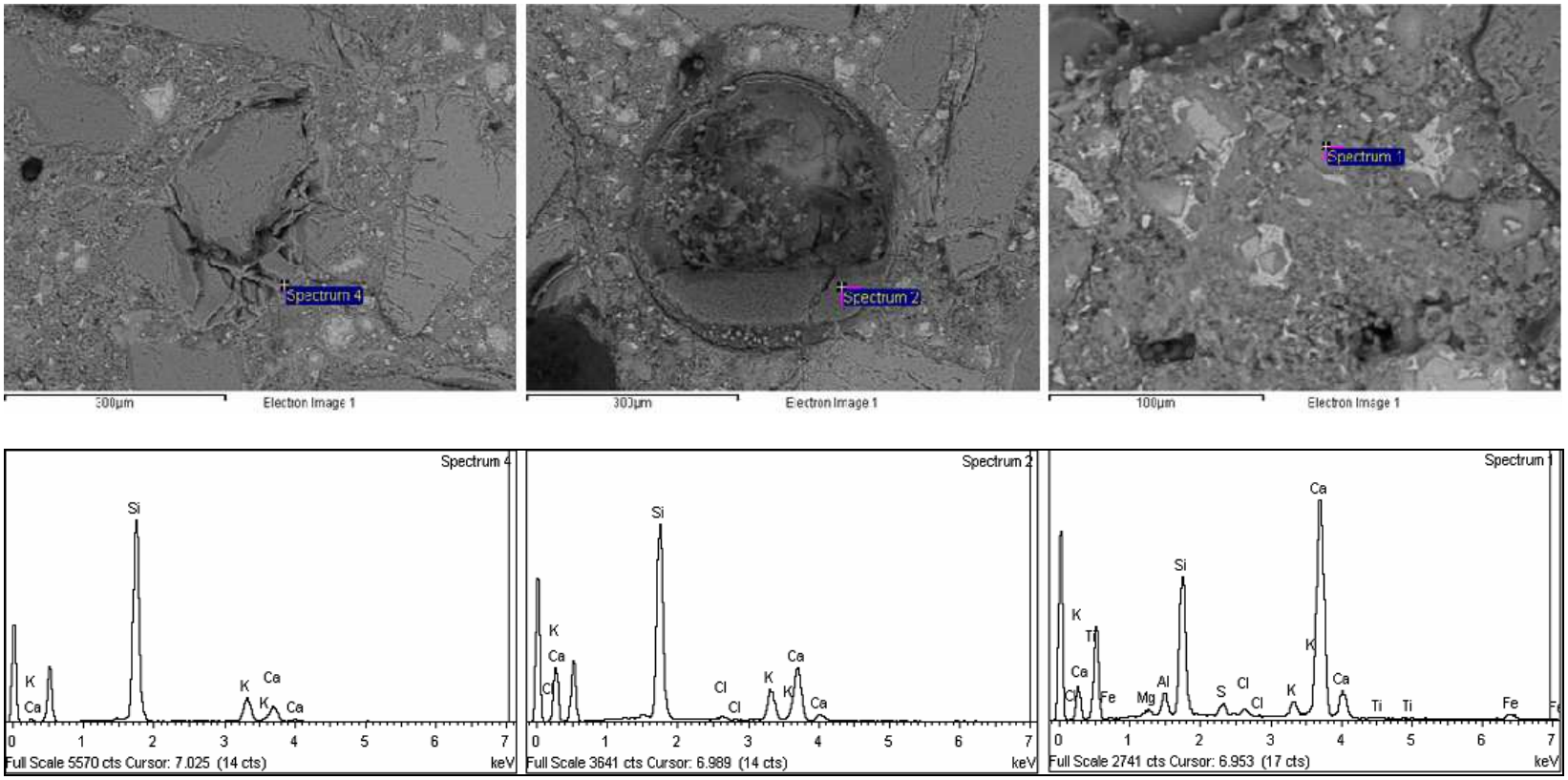
Alkali/ Silica = 0.27
Calcium/ Silica = 0.26

Alkali/ Silica = 0.30
Calcium/ Silica = 1.03

Alkali/ Silica = Nil
Calcium/ Silica = 2.30
% Chloride = 1.94

Figure 5.18: SEM/ EDX analysis results: Mortar specimens subjected to 3% Potassium chloride solution

c) Type I Low alkali cement, 80°C



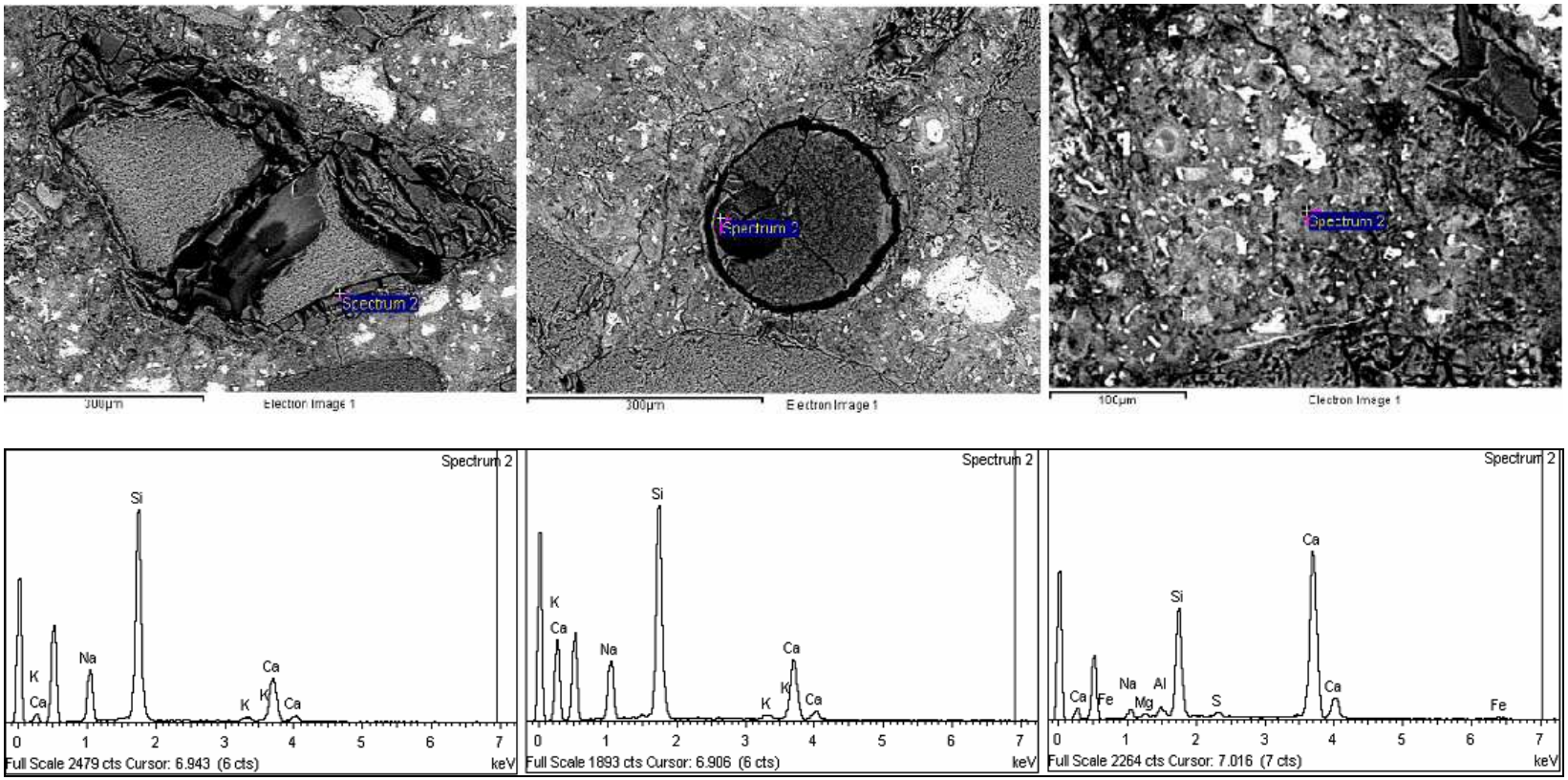
Alkali/ Silica = 0.23
Calcium/ Silica = 0.16

Alkali/ Silica = 0.29
Calcium/ Silica = 0.58

Alkali/ Silica = 0.15
Calcium/ Silica = 2.61
% Chloride = 1.44

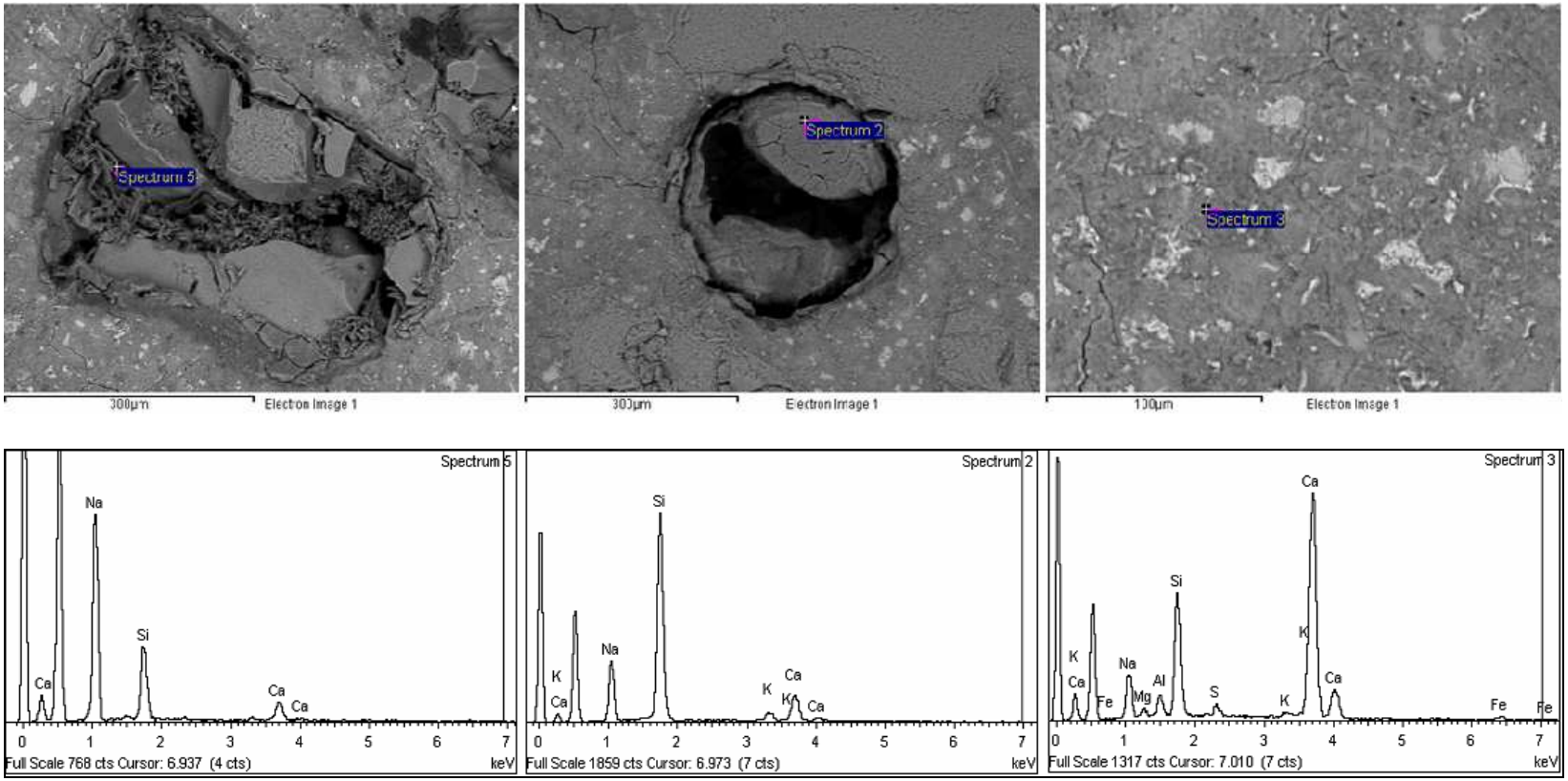
Figure 5.18: SEM/ EDX analysis results: Mortar specimens subjected to 3% Potassium chloride solution

d) Type I High alkali cement, 80°C



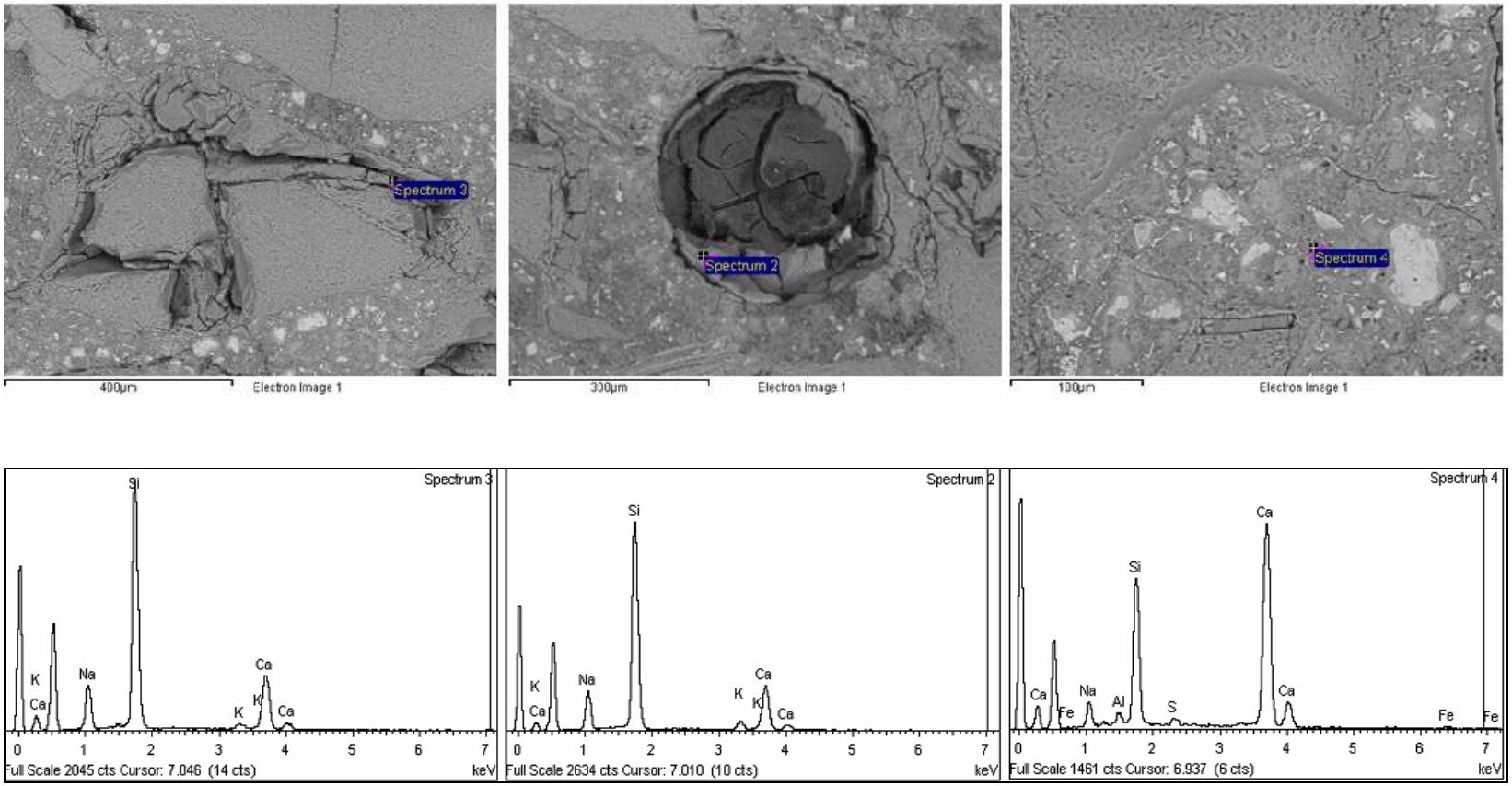
Alkali/ Silica = 0.32	Alkali/ Silica = 0.37	Alkali/ Silica = 0.15
Calcium/ Silica = 0.40	Calcium/ Silica = 0.52	Calcium/ Silica = 2.60

Figure 5.19: SEM/ EDX analysis results: Mortar specimens subjected to 1N Sodium hydroxide solution
a) Type I Low alkali cement, 38°C



Alkali/ Silica = 1.83	Alkali/ Silica = 0.38	Alkali/ Silica = 0.60
Calcium/ Silica = 0.32	Calcium/ Silica = 0.23	Calcium/ Silica = 2.90

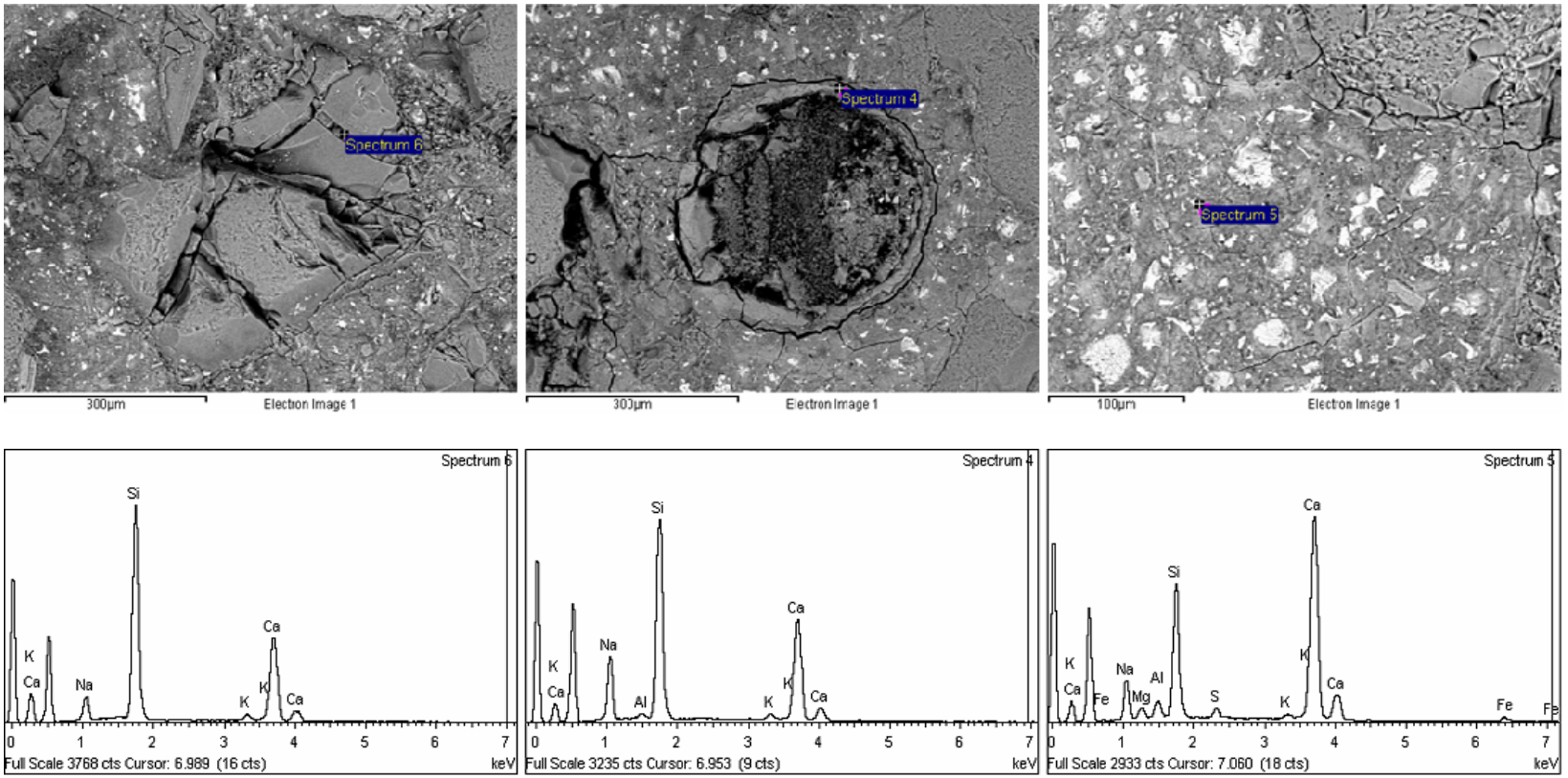
Figure 5.19: SEM/ EDX analysis results: Mortar specimens subjected to 1N Sodium hydroxide solution
b) Type I High alkali cement, 38°C



Alkali/ Silica = 0.25	Alkali/ Silica = 0.29	Alkali/ Silica = 0.26
Calcium/ Silica = 0.41	Calcium/ Silica = 0.41	Calcium/ Silica = 2.27

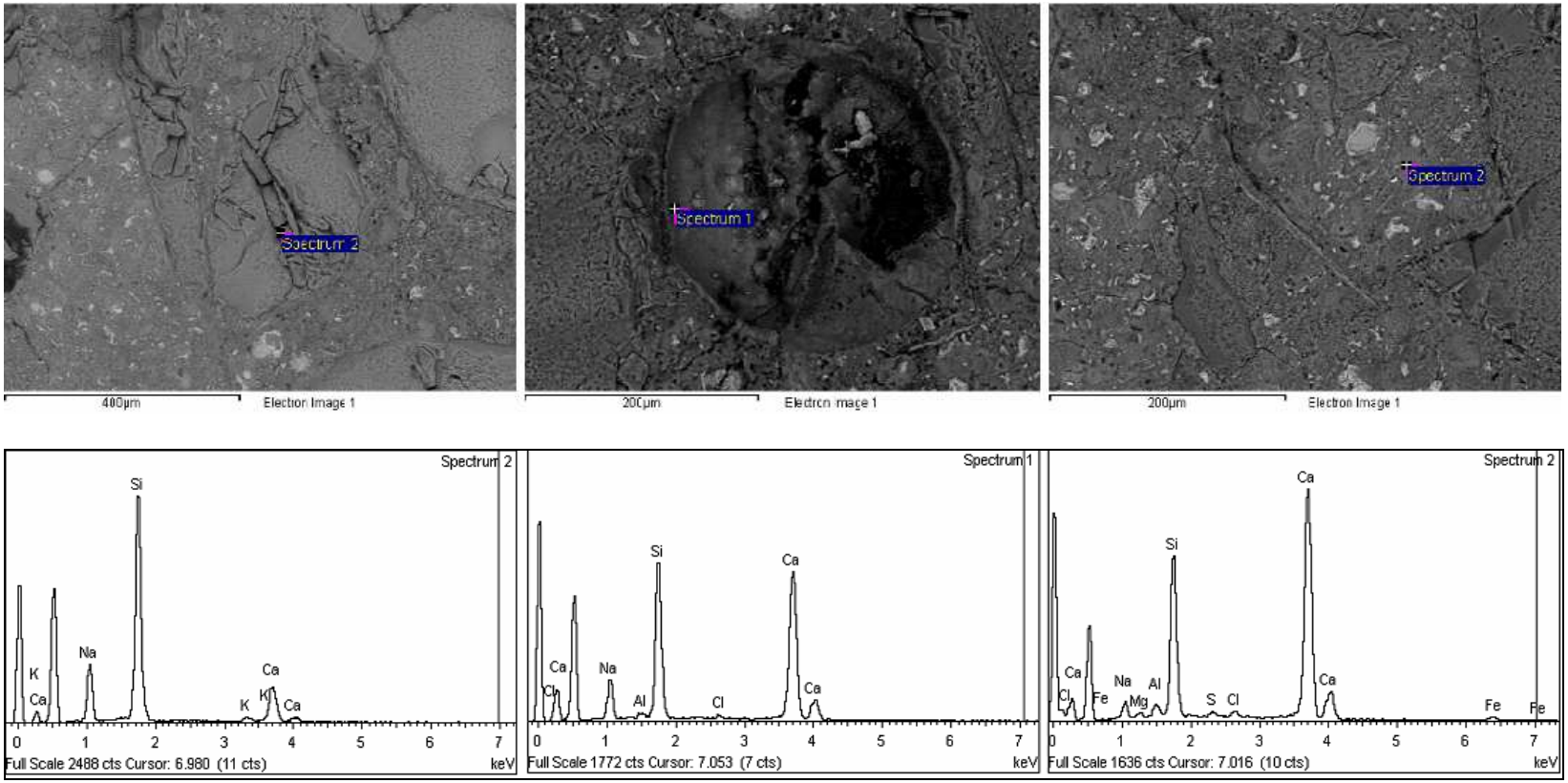
Figure 5.19: SEM/ EDX analysis results: Mortar specimens subjected to 1N Sodium hydroxide solution

c) Type I Low alkali cement, 80°C



Alkali/ Silica = 0.20	Alkali/ Silica = 0.44	Alkali/ Silica = 0.49
Calcium/ Silica = 0.75	Calcium/ Silica = 0.86	Calcium/ Silica = 2.32

Figure 5.19: SEM/ EDX analysis results: Mortar specimens subjected to 1N Sodium hydroxide solution
d) Type I High alkali cement, 80°C



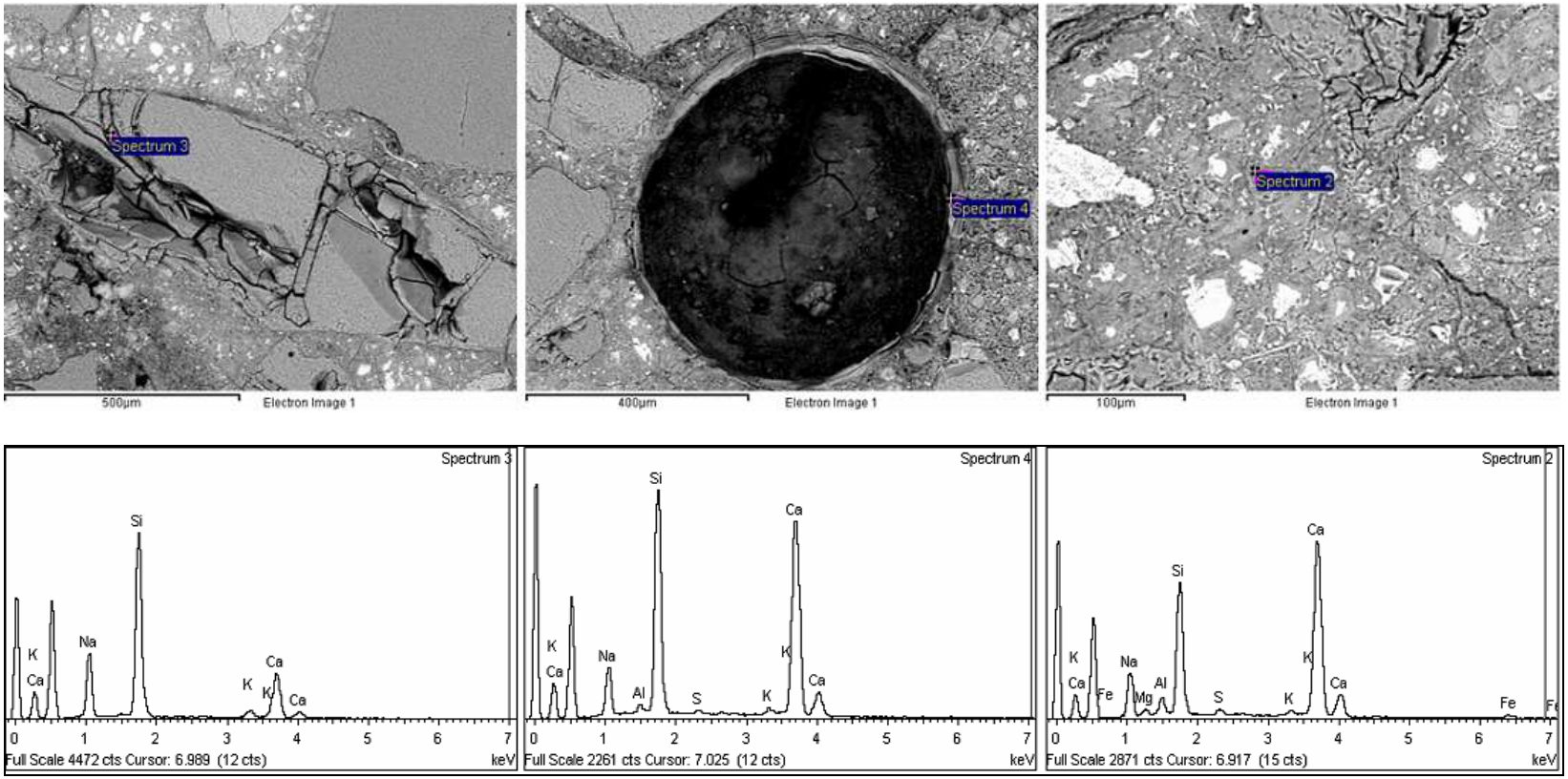
Alkali/ Silica = 0.32
Calcium/ Silica = 0.29

Alkali/ Silica = 0.39
Calcium/ Silica = 1.64

Alkali/ Silica = 0.16
Calcium/ Silica = 2.33
% Chloride = 1.60

Figure 5.20: SEM/ EDX analysis results: Mortar specimens subjected to 0.5N Sodium hydroxide and 3% Sodium chloride blended solution

a) Type I Low alkali cement, 38°C



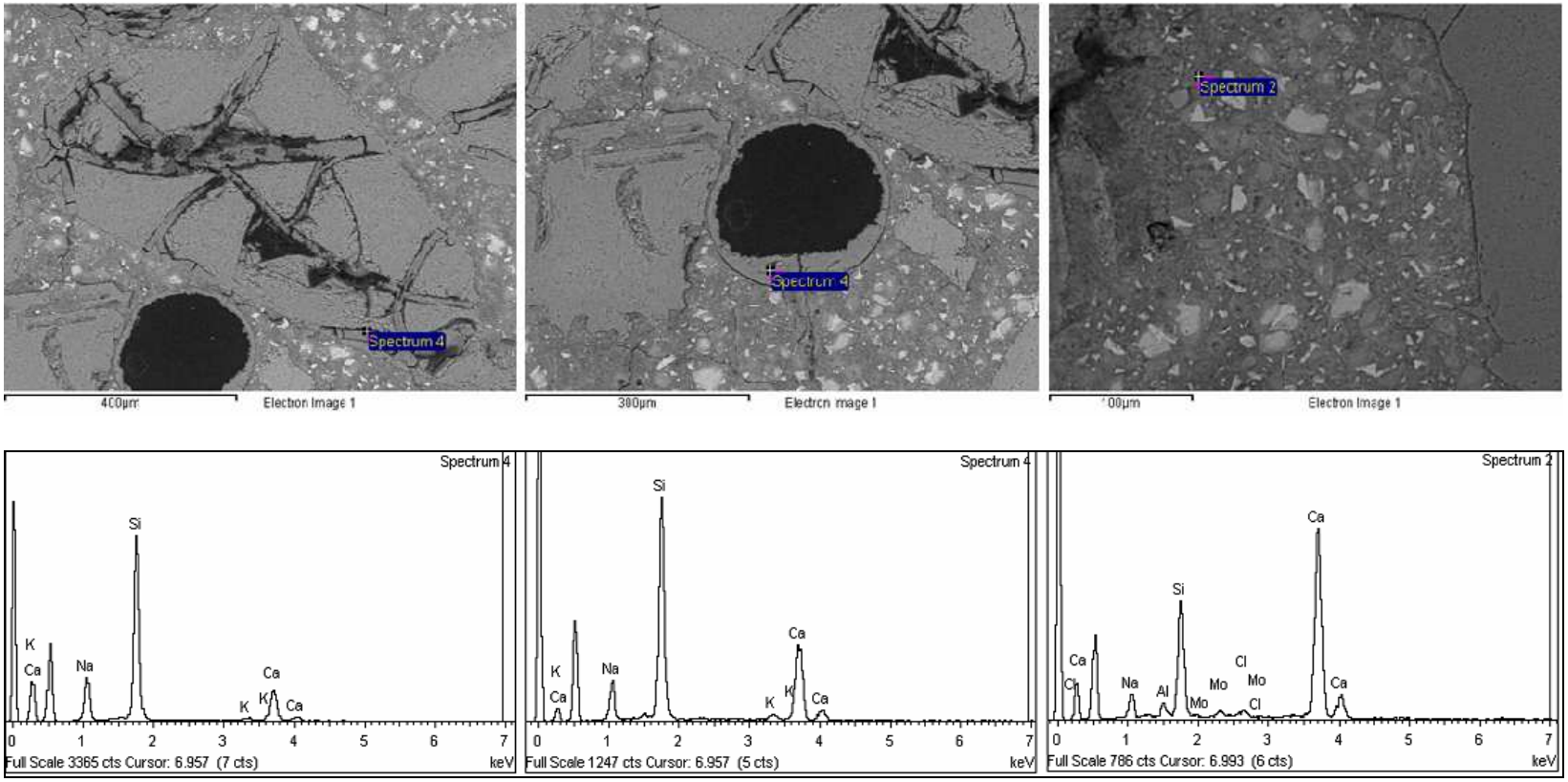
Alkali/ Silica = 0.45
Calcium/ Silica = 0.44

Alkali/ Silica = 0.35
Calcium/ Silica = 1.53

Alkali/ Silica = 0.52
Calcium/ Silica = 2.13
% Chloride = -

Figure 5.20: SEM/ EDX analysis results: Mortar specimens subjected to 0.5N Sodium hydroxide and 3% Sodium chloride blended solution

b) Type I High alkali cement, 38°C



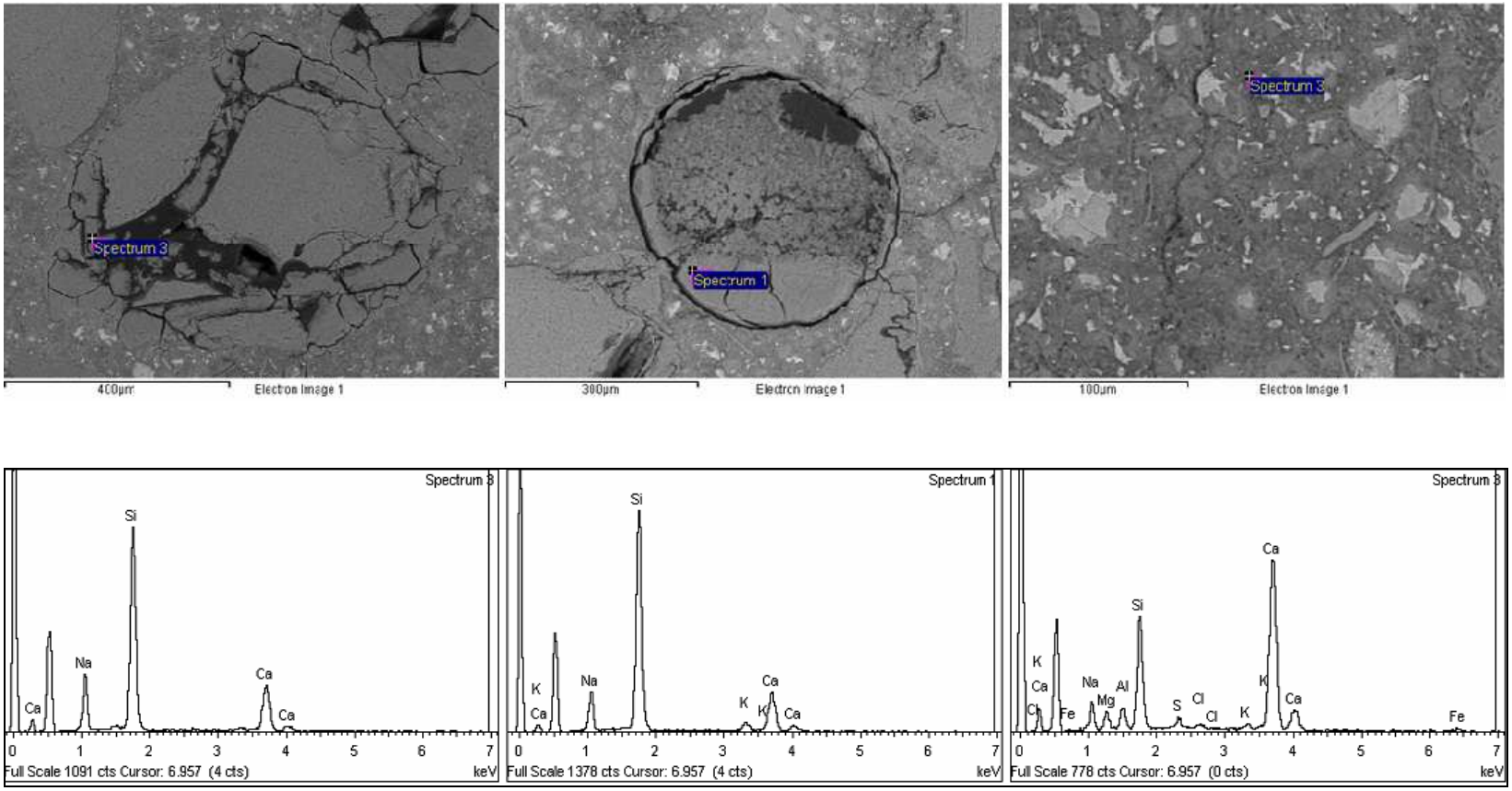
Alkali/ Silica = 0.29
Calcium/ Silica = 0.33

Alkali/ Silica = 0.27
Calcium/ Silica = 0.68

Alkali/ Silica = 0.33
Calcium/ Silica = 2.82
% Chloride = 1.43

Figure 5.20: SEM/ EDX analysis results: Mortar specimens subjected to 0.5N Sodium hydroxide and 3% Sodium chloride blended solution

c) Type I Low alkali cement, 80°C



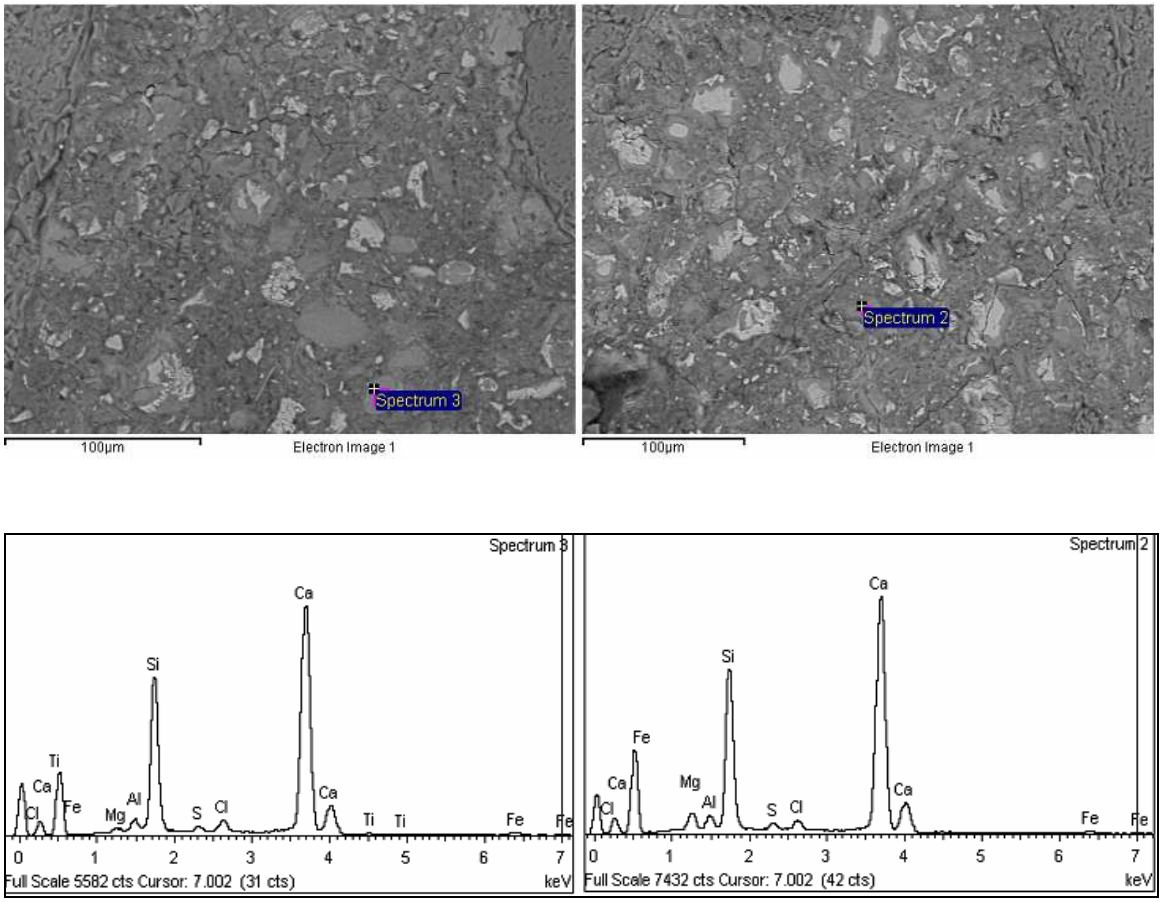
Alkali/ Silica = 0.31
Calcium/ Silica = 0.42

Alkali/ Silica = 0.28
Calcium/ Silica = 0.35

Alkali/ Silica = 0.38
Calcium/ Silica = 2.40
% Chloride = 1.20

Figure 5.20: SEM/ EDX analysis results: Mortar specimens subjected to 0.5N Sodium hydroxide and 3% Sodium chloride blended solution

d) Type I Low alkali cement, 80°C



a) % Chloride = 2.74 b) % Chloride= 2.08

Figure 5.21: SEM/ EDX analysis results: Mortar specimens subjected to 3% Magnesium chloride solution

a) Type I Low alkali cement, 80°C
b) Type I High alkali cement, 80°C

CHAPTER SIX

SUMMARY AND CONCLUSIONS

6.1 General

This chapter summarizes the results of Standard ASTM C 1260 tests, Mortar Bar tests, Titrations, ICP tests, X-ray Diffraction studies and SEM/ EDX analyses. All the results are correlated to conclude the research in line with the research objectives. Results summarized here are in context of mortar bars using fused silica as reactive aggregate.

6.2 Standard ASTM C 1260, Mortar Bar test

These tests served as the stepping stone to determine potential of chloride deicers to cause alkali silica reaction in concrete under the effect of variables like temperature and cement. Findings of these tests can be summarized as follows,

1. Mortar bars using fused silica as aggregate expanded more than 0.1% at 14 days in the standard ASTM C 1260 test
2. Mortar specimens subjected to alkali chloride solutions expanded more than those subjected to 1N sodium hydroxide solution in the standard ASTM C 1260 test
3. Expansion in mortar bars exposed to blended solution of 0.5N sodium hydroxide and 3% Sodium chloride solution was more than mortar bars exposed to 1N sodium hydroxide solution
4. Mortar bars in calcium chloride/ magnesium chloride solutions expanded less than those in sodium- chloride/ potassium chloride solutions

5. Expansion in mortar bars exposed to calcium chloride solution and magnesium chloride solution were comparable to the expansion observed in mortar bars exposed to de-ionized water
6. Expansion of mortar bars was more in potassium chloride solution than in sodium chloride solution at lower temperature. However, at high temperature sodium chloride caused higher expansion than potassium chloride solution
7. Rate of expansion of mortar bars was fairly constant in the test duration in mortar bars exposed to alkali-chloride solutions
8. Expansions in mortars bars subject to alkali chloride solution were comparable while using cements of different alkali contents.

6.3 X-ray Diffraction

This test involved use of specimens prepared by 50% replacement of natural sand with fused silica, Type I Low alkali cement. They were exposed to the solution at a temperature of 38°C for a period of 160 days. Findings of this test can be summarized as follows:

1. Chloroaluminate, also known as Friedel's salt, was identified in mortar specimens subjected to sodium chloride, calcium chloride and potassium chloride solution. They were absent in specimens subjected to de-ionized water. X-ray diffraction diagram also showed peaks of ettringite in all solutions
2. Distinct peaks of calcium hydroxide were observed in X-ray diffraction of specimens subjected to de-ionized water and calcium chloride solutions. Alkali chlorides react

with calcium hydroxide to form alkalis, hydroxyl ions and calcium chloride. Calcium chloride further reacts with ettringite to form chloroaluminates

3. Intensity of Friedel's salt peaks was more intense in mortar specimens exposed to potassium chloride solution compared to those exposed to sodium chloride solution.

6.4 Titrations

Findings of this test can be summarized as follows,

1. Hydroxyl ion concentration in solution filtered from crushed mortar subjected to alkali chloride solutions was very low compared to solution filtered from crushed mortar subjected to alkali hydroxide solution. This means that ASR effect should be less in mortar bars exposed to alkali chlorides. This contradicts with findings of Mortar Bar test

6.5 SEM/ EDX analysis

Findings of this test can be summarized as follows,

1. Mortar samples exposed to de-ionized water, calcium chloride and magnesium chloride showed no signs of cracks irrespective of the temperature and type of cement used
2. ASR gel was observed in and around fused silica aggregate, in air voids and in surrounding matrix in case of mortar bars subjected to alkali chloride/ alkali hydroxide solutions. Cement matrix showed significant number of micro-cracks resulting from ASR damage and probably also shrinkage during sample preparation.

6.6 Influence of selected factors

1. Effect of Aggregate

Standard ASTM C 1260 results confirm that fused silica is a highly reactive aggregate. This is supported by the fact that expansion produced by fused silica bearing mortar bars is very high compared to expansion produced by natural sand bearing mortar bars under all conditions

2. Effect of Temperature

Standard ASTM C 1260 and Mortar Bar test results show that mortar bars exposed to alkali chloride/ alkali hydroxide solutions at 80°C expand more than those exposed to similar solutions at 38°C

3. Effect of Type of Cement

Figure (5.9) shows that cement type seems to have little influence on expansion of mortar specimens subjected to external alkali solutions

6.7 Conclusion

1. Effect of Chloride deicers on Alkali silica reaction

Sodium chloride and potassium chloride accelerate ASR in concrete. Calcium chloride and magnesium chloride seem to cause minimal ASR effect. Typically alkali silica gel is rich in alkalis and silica. Increase in calcium concentration in the gel leads to the formation of a non-swelling gel. This is supported by findings from Standard ASTM C 1260, Mortar bar test and SEM/EDX analysis results. Potassium ions accelerate the ASR reaction in comparison to sodium ions at lower temperatures.

SEM/EDX results show that calcium concentration in ASR gel increases with distance from aggregate. During the ASR reaction, some of the gel migrates to the matrix surrounding the aggregate. This gel on coming in contact with cement hydration products becomes rich in calcium. This gel further propagates the ASR reaction by increasing the alkali availability to more reaction sites.

Expansion results imply that chlorides play an important role in accelerating the ASR reaction. Chloride ions by itself do not contribute to the expansive effect. This can be concluded based on the fact that sodium chloride causes more expansion than calcium chloride solution.

2. Alkali silica reaction caused by chloride deicers

Mortar bar test results show that alkali chlorides cause ASR. SEM/EDX analysis results of mortar specimens subject to alkali chloride solutions show chlorides dispersed in the cement matrix. This suggests the interaction of chloride ions with cement hydration products to form chloroaluminates. Presence of chloroaluminates in such specimens was confirmed by X-ray diffraction results.

Alkali chlorides react with calcium hydroxide to form alkali hydroxides and calcium chloride. Calcium chloride reacts with ettringite to form Friedel's salt or chloroaluminates where the sulfate ions are replaced by chloride ions. Thus, the formation of Friedel's salt releases the alkalis and hydroxyl ions into the system. They further react with reactive silica to form alkali silica gel. Low intensity peaks of chloroaluminates in X-ray diffraction results suggest that the transformation of ettringite to Friedel's salt is a timely process.

3. Effect of Temperature

ASR is accelerated at higher temperature in presence of alkali chlorides. The effect is confirmed by expansion results and visual inspection

4. Effect of type of cement

Type of cement does not have much influence on expansion results, particularly in case of presence of external alkalis. Although high-alkali cement based mortar bars show higher alkali/silica ratio in ASR gel

5. Effect of type of aggregate

Mortar bars with fused silica (a reactive aggregate) expand more than mortar bars with natural sand (a non-reactive aggregate).

The knowledge acquired by this research can help establish better and effective conditions to form a more reliable and accelerated alternate test method.

6.8 Recommendations for future work

Expansion results for mortar specimens subjected to de-ionized water, calcium chloride and magnesium chloride are more at 38°C than at 80°C. This contradicts with expansive behavior in mortar specimens subject to alkali chloride solutions. It can be hypothesized that the low expansion at 80°C is due to the reduced alkali/ silica ratio. At higher temperature the calcium ions may have diffused faster to the reaction sites leading to reduced expansion. This aspect needs further study to arrive at a conclusion.

ICP test and Titration results do not show any specific trend to arrive at a conclusion. They are not conclusive enough and need further investigation.

Expansion tests need to be carried out using a real aggregate like Spratt.

APPENDICES

Appendix A:

LENGTH CHANGE OF MORTAR BARS

Table A.1 Expansion results of mortar bars using natural sand/ (50% fused silica+ 50% natural sand), Type I Low/ High alkali cement subjected to de-ionized water at 38°C

Days	S-LC at 38°C		FS-LC at 38°C		S-HC at 38°C		FS-HC at 38°C	
	Exp. (%)	Std. Dev.	Exp. (%)	Std. Dev.	Exp. (%)	Std. Dev.	Exp. (%)	Std. Dev.
0	0.00	0.000	0.00	0.000	0.00	0.000	0.00	0.000
3	0.00	0.002	0.03	0.003	0.00	0.000	0.10	0.001
7	0.00	0.002	0.07	0.005	0.01	0.001	0.20	0.003
10	0.00	0.001	0.08	0.005	0.01	0.000	0.23	0.008
14	0.00	0.001	0.08	0.004	0.01	0.000	0.24	0.011
21	0.00	0.001	0.08	0.005	0.01	0.000	0.24	0.013
28	0.00	0.001	0.08	0.005	0.01	0.001	0.24	0.013
35	0.00	0.002	0.08	0.006	0.01	0.001	0.25	0.012
42	0.00	0.004	0.08	0.001	0.01	0.001	0.25	0.013
49	0.00	0.004	0.08	0.000	0.01	0.004	0.25	0.013
56	0.00	0.004	0.08	0.003	0.01	0.002	0.25	0.013
63	0.00	0.002	0.08	0.006	0.01	0.001	0.24	0.013
70	0.00	0.003	0.08	0.007	0.01	0.001	0.24	0.013
77	0.01	0.002	0.08	0.007	0.01	0.001	0.25	0.011
84	0.01	0.001	0.09	0.006	0.02	0.004	0.25	0.011
91	0.01	0.001	0.09	0.006	0.02	0.001	0.25	0.013
98	0.01	0.001	0.08	0.009	0.01	0.001	0.25	0.019
105	0.01	0.003	0.08	0.006	0.02	0.001	0.25	0.013
112	0.01	0.006	0.08	0.008	0.01	0.001	0.25	0.015
119	0.00	0.005	0.08	0.008	0.01	0.001	0.24	0.015
126	0.00	0.002	0.08	0.008	0.01	0.001	0.26	0.013
133	0.00	0.003	0.07	0.007	0.01	0.001	0.25	0.016
140	0.00	0.002	0.08	0.007	0.02	0.001	0.25	0.016
147	0.00	0.009	0.07	0.007	0.01	0.008	0.25	0.017
154	0.01	0.008	0.08	0.006	0.02	0.001	0.26	0.005
161	0.01	0.004	0.08	0.012	0.02	0.000	0.26	0.013

Table A.2 Expansion results of mortar bars using natural sand/ (50% fused silica+ 50% natural sand), Type I Low/ High alkali cement subjected to 3% Sodium chloride solution at 38°C

Days	S-LC at 38°C		FS-LC at 38°C		S-HC at 38°C		FS-HC at 38°C	
	Exp. (%)	Std. Dev.	Exp. (%)	Std. Dev.	Exp. (%)	Std. Dev.	Exp. (%)	Std. Dev.
0	0.00	0.000	0.00	0.000	0.00	0.000	0.00	0.000
3	0.01	0.001	0.05	0.004	0.01	0.002	0.12	0.004
7	0.01	0.006	0.12	0.004	0.01	0.002	0.31	0.008
10	0.01	0.006	0.20	0.007	0.01	0.002	0.31	0.008
14	0.01	0.004	0.23	0.007	0.02	0.005	0.39	0.021
21	0.00	0.005	0.39	0.014	0.00	0.003	0.50	0.010
28	0.04	0.060	0.50	0.013	0.02	0.002	0.61	0.010
35	0.01	0.005	0.64	0.029	0.02	0.004	0.74	0.021
42	0.01	0.006	0.76	0.017	0.02	0.002	0.85	0.013
49	0.01	0.006	0.86	0.032	0.02	0.003	0.94	0.005
56	0.01	0.005	1.00	0.022	0.02	0.001	1.10	0.019
63	0.01	0.006	1.10	0.039	0.03	0.001	1.18	0.020
70	0.01	0.005	1.20	0.031	0.03	0.002	1.26	0.018
77	0.01	0.007	1.30	0.036	0.03	0.001	1.36	0.019
84	0.01	0.007	1.38	0.043	0.03	0.002	1.45	0.019
91	0.01	0.006	1.48	0.044	0.03	0.002	1.54	0.018
98	0.01	0.015	1.58	0.042	0.03	0.000	1.63	0.021
105	0.02	0.015	1.68	0.045	0.03	0.000	1.72	0.023
112	0.01	0.015	1.78	0.042	0.03	0.001	1.82	0.025
119	0.00	0.013	1.89	0.043	0.03	0.000	1.92	0.030
126	0.01	0.008	1.98	0.059	0.03	0.003	1.97	0.074
133	0.01	0.006	2.04	0.037	0.03	0.002	2.03	0.032
140	0.01	0.006	2.13	0.046	0.03	0.001	2.13	0.039
147	0.01	0.008	2.21	0.074	0.04	0.004	2.21	0.069

Table A.3 Expansion results of mortar bars using natural sand/ (50% fused silica+ 50% natural sand), Type I Low/ High alkali cement subjected to 3% Calcium chloride solution at 38°C

Days	S-LC at 38°C		FS-LC at 38°C		S-HC at 38°C		FS-HC at 38°C	
	Exp. (%)	Std. Dev.	Exp. (%)	Std. Dev.	Exp. (%)	Std. Dev.	Exp. (%)	Std. Dev.
0	0.00	0.000	0.00	0.000	0.00	0.000	0.00	0.000
3	0.01	0.004	0.04	0.003	0.01	0.001	0.08	0.023
7	0.01	0.004	0.06	0.004	0.01	0.001	0.17	0.005
10	0.01	0.002	0.06	0.004	0.02	0.000	0.18	0.005
14	0.01	0.002	0.05	0.006	0.01	0.001	0.18	0.005
21	0.01	0.006	0.06	0.003	0.02	0.001	0.19	0.004
28	0.01	0.006	0.06	0.003	0.02	0.001	0.19	0.004
35	0.01	0.006	0.06	0.003	0.02	0.001	0.19	0.004
42	0.02	0.005	0.07	0.003	0.03	0.001	0.20	0.005
49	0.01	0.005	0.06	0.003	0.03	0.001	0.20	0.004
56	0.02	0.006	0.06	0.003	0.03	0.001	0.20	0.004
63	0.01	0.006	0.06	0.003	0.03	0.000	0.20	0.004
70	0.01	0.003	0.07	0.003	0.03	0.000	0.20	0.005
77	0.01	0.002	0.07	0.004	0.03	0.001	0.20	0.006
84	0.01	0.059	0.06	0.004	0.03	0.001	0.20	0.007
91	0.01	0.011	0.07	0.004	0.03	0.001	0.20	0.000
98	0.01	0.002	0.07	0.004	0.03	0.001	0.20	0.006
105	0.02	0.002	0.07	0.003	0.04	0.000	0.21	0.008
112	0.02	0.012	0.06	0.004	0.04	0.002	0.20	0.001
119	0.01	0.000	0.06	0.003	0.03	0.001	0.20	0.023
126	0.01	0.001	0.07	0.001	0.04	0.002	0.20	0.006
133	0.01	0.003	0.07	0.004	0.04	0.001	0.21	0.004
140	0.02	0.000	0.07	0.003	0.04	0.001	0.21	0.004
147	0.01	0.006	0.07	0.001	0.04	0.003	0.20	0.004
154	0.02	0.002	0.07	0.059	0.04	0.004	0.21	0.006
161	0.02	0.000	0.07	0.001	0.04	0.003	0.21	0.004

Table A.4 Expansion results of mortar bars using natural sand/ (50% fused silica+ 50% natural sand), Type I Low/ High alkali cement subjected to 3% Potassium chloride solution at 38°C

Days	S-LC at 38°C		FS-LC at 38°C		S-HC at 38°C		FS-HC at 38°C	
	Exp. (%)	Std. Dev.	Exp. (%)	Std. Dev.	Exp. (%)	Std. Dev.	Exp. (%)	Std. Dev.
0	0.00	0.000	0.00	0.000	0.00	0.000	0.00	0.000
3	0.00	0.001	0.05	0.003	0.01	0.000	0.11	0.003
7	0.01	0.001	0.18	0.005	0.01	0.000	0.26	0.001
10	0.01	0.000	0.26	0.006	0.02	0.001	0.34	0.009
14	0.01	0.001	0.38	0.009	0.02	0.000	0.44	0.011
21	0.01	0.001	0.57	0.010	0.02	0.000	0.61	0.013
28	0.02	0.001	0.70	0.006	0.02	0.001	0.73	0.017
35	0.02	0.001	0.83	0.015	0.02	0.001	0.86	0.007
42	0.02	0.001	0.96	0.008	0.03	0.000	0.99	0.010
49	0.02	0.001	1.08	0.013	0.03	0.001	1.09	0.005
56	0.02	0.003	1.21	0.044	0.03	0.001	1.18	0.006
63	0.01	0.004	1.36	0.049	0.02	0.001	1.32	0.008
70	0.01	0.004	1.44	0.038	0.03	0.001	1.39	0.021
77	0.02	0.001	1.55	0.030	0.03	0.000	1.48	0.015
84	0.02	0.001	1.69	0.034	0.03	0.000	1.62	0.023
91	0.02	0.001	1.84	0.044	0.03	0.001	1.73	0.020
98	0.02	0.001	1.94	0.055	0.03	0.001	1.83	0.015
105	0.02	0.003	2.02	0.033	0.03	0.001	1.94	0.026
112	0.02	0.003	2.18	0.030	0.03	0.002	2.08	0.009
119	0.02	0.004	2.27	0.016	0.03	0.000	2.16	0.009
126	0.02	0.001	2.36	0.018	0.04	0.001	2.25	0.010
133	0.01	0.002	2.45	0.034	0.03	0.002	2.34	0.005
140	0.03	0.001	2.60	0.027	0.04	0.001	2.43	0.028
147	0.02	0.001	2.60	0.000	0.03	0.001	2.49	0.000
154	0.02	0.004	2.67	0.000	0.04	0.005	2.55	0.002
161	0.01	0.001	2.76	0.000	0.03	0.001	2.64	0.000

Table A.5 Expansion results of mortar bars using natural sand/ (50% fused silica+ 50% natural sand), Type I Low/ High alkali cement subjected to 1N Sodium hydroxide solution at 38°C

Days	S-LC at 38°C		FS-LC at 38°C		S-HC at 38°C		FS-HC at 38°C	
	Exp. (%)	Std. Dev.	Exp. (%)	Std. Dev.	Exp. (%)	Std. Dev.	Exp. (%)	Std. Dev.
0	0.00	0.000	0.00	0.000	0.00	0.000	0.00	0.000
3	0.00	0.001	0.12	0.024	0.00	0.000	0.13	0.004
7	0.00	0.001	0.32	0.027	0.01	0.000	0.33	0.008
10	0.00	0.001	0.39	0.028	0.01	0.001	0.40	0.008
14	0.00	0.001	0.51	0.026	0.01	0.000	0.51	0.008
21	0.00	0.001	0.71	0.022	0.01	0.001	0.70	0.014
28	0.00	0.001	0.82	0.021	0.01	0.002	0.80	0.018
35	0.04	0.053	0.94	0.022	0.01	0.002	0.90	0.020
42	0.01	0.001	1.01	0.026	0.01	0.002	0.96	0.025
49	0.00	0.007	1.08	0.031	0.02	0.001	1.02	0.025
56	0.01	0.000	1.13	0.034	0.00	0.002	1.06	0.027
63	0.01	0.001	1.21	0.038	0.02	0.001	1.14	0.026
70	0.01	0.003	1.26	0.040	0.02	0.002	1.18	0.027
77	0.01	0.001	1.31	0.045	0.02	0.002	1.23	0.031
84	0.01	0.001	1.37	0.044	0.02	0.001	1.29	0.029
91	0.00	0.001	1.41	0.048	0.02	0.002	1.32	0.038
98	0.01	0.003	1.44	0.049	0.01	0.001	1.35	0.035
105	0.01	0.001	1.49	0.052	0.02	0.002	1.40	0.036
112	0.01	0.001	1.53	0.055	0.02	0.001	1.44	0.037
119	0.00	0.001	1.56	0.057	0.02	0.001	1.46	0.035
126	0.00	0.001	1.58	0.059	0.01	0.002	1.49	0.037
133	0.00	0.003	1.61	0.060	0.02	0.006	1.52	0.038
140	0.01	0.002	1.66	0.064	0.02	0.002	1.56	0.036
147	0.00	0.004	1.68	0.063	0.01	0.001	1.58	0.038
154	0.00	0.003	1.71	0.065	0.02	0.002	1.61	0.040
161	0.00	0.004	1.74	0.066	0.02	0.003	1.63	0.037

Table A.6 Expansion results of mortar bars using natural sand/ (50% fused silica+ 50% natural sand), Type I Low/ High alkali cement subjected to 0.5N Sodium hydroxide and 3 % Sodium chloride blended solution at 38°C

Days	S-LC at 38°C		FS-LC at 38°C		S-HC at 38°C		FS-HC at 38°C	
	Exp. (%)	Std. Dev.	Exp. (%)	Std. Dev.	Exp. (%)	Std. Dev.	Exp. (%)	Std. Dev.
0	0.00	0.000	0.00	0.000	0.00	0.000	0.00	0.000
3	0.00	0.002	0.10	0.001	0.00	0.002	0.15	0.007
7	0.01	0.001	0.26	0.009	0.01	0.002	0.31	0.011
10	0.01	0.001	0.40	0.011	0.01	0.004	0.45	0.017
14	0.01	0.005	0.52	0.016	0.01	0.005	0.56	0.019
21	0.00	0.008	0.77	0.028	0.01	0.012	0.80	0.020
28	0.01	0.005	0.93	0.035	0.01	0.012	0.94	0.021
35	0.00	0.006	1.10	0.036	0.01	0.012	1.09	0.019
42	0.00	0.010	1.35	0.036	0.01	0.010	1.31	0.027
49	0.00	0.012	1.49	0.032	0.01	0.010	1.42	0.026
56	0.00	0.000	1.55	0.000	0.01	0.000	1.47	0.000
63	0.01	0.000	1.62	0.034	0.01	0.010	1.52	0.022
70	0.01	0.004	1.73	0.034	0.02	0.010	1.62	0.027
77	0.01	0.006	1.86	0.039	0.02	0.008	1.73	0.030
84	0.01	0.007	1.93	0.034	0.02	0.011	1.80	0.030
91	0.01	0.003	1.98	0.026	0.02	0.008	1.86	0.029
98	0.01	0.003	2.02	0.081	0.02	0.014	1.93	0.033
105	0.01	0.004	2.15	0.033	0.01	0.009	1.99	0.037
112	0.02	0.002	2.24	0.028	0.02	0.010	2.06	0.036
119	0.00	0.004	2.28	0.032	0.01	0.012	2.09	0.037
126	0.01	0.002	2.36	0.031	0.02	0.009	2.15	0.035
133	0.01	0.012	2.41	0.030	0.02	0.017	2.19	0.038
140	0.01	0.009	2.47	0.034	0.00	0.010	2.24	0.039
147	0.00	0.012	2.55	0.027	0.01	0.010	2.30	0.037
154	0.01	0.008	2.60	0.034	0.02	0.010	2.36	0.039
161	0.00	0.007	2.62	0.000	0.01	0.010	2.39	0.039

Table A.7 Expansion results of mortar bars using natural sand/ (50% fused silica+ 50% natural sand), Type I Low/ High alkali cement subjected to 3% Magnesium chloride solution at 38°C

Days	S-LC at 38°C		FS-LC at 38°C		S-HC at 38°C		FS-HC at 38°C	
	Exp. (%)	Std. Dev.	Exp. (%)	Std. Dev.	Exp. (%)	Std. Dev.	Exp. (%)	Std. Dev.
0	0.00	0.000	0.00	0.000	0.00	0.000	0.00	0.000
3	0.00	0.003	0.03	0.001	0.00	0.121	0.07	0.002
7	0.01	0.005	0.05	0.007	0.01	0.005	0.12	0.001
10	0.00	0.004	0.05	0.006	0.00	0.004	0.13	0.002
14	0.01	0.008	0.05	0.005	0.01	0.007	0.13	0.002
21	0.01	0.010	0.04	0.005	0.00	0.006	0.13	0.004
28	0.00	0.005	0.05	0.010	0.00	0.007	0.13	0.002
35	0.00	0.005	0.05	0.003	0.01	0.006	0.13	0.001
42	0.00	0.006	0.04	0.005	0.01	0.013	0.13	0.004
49	0.00	0.005	0.05	0.005	0.01	0.008	0.14	0.006
56	0.01	0.002	0.04	0.004	0.01	0.008	0.14	0.003
63	0.02	0.005	0.05	0.003	0.02	0.009	0.15	0.002
70	0.02	0.002	0.05	0.004	0.02	0.008	0.15	0.003
77	0.02	0.003	0.05	0.004	0.02	0.006	0.15	0.004
84	0.02	0.004	0.06	0.004	0.03	0.008	0.15	0.003
91	0.02	0.006	0.05	0.003	0.03	0.010	0.16	0.004
98	0.02	0.005	0.05	0.004	0.02	0.007	0.15	0.004
105	0.02	0.005	0.06	0.004	0.03	0.008	0.15	0.001
112	0.02	0.003	0.06	0.004	0.03	0.008	0.16	0.002
119	0.02	0.005	0.06	0.007	0.03	0.008	0.16	0.003
126	0.02	0.005	0.06	0.003	0.03	0.007	0.16	0.002
133	0.02	0.003	0.06	0.004	0.04	0.012	0.16	0.005

Table A.8 Expansion results of mortar bars using natural sand/ (50% fused silica+ 50% natural sand), Type I Low/ High alkali cement subjected to de-ionized water at 80°C

Days	S-LC at 80°C		FS-LC at 80°C		S-HC at 80°C		FS-HCat 80°C	
	Exp. (%)	Std. Dev.	Exp. (%)	Std. Dev.	Exp. (%)	Std. Dev.	Exp. (%)	Std. Dev.
0	0.00	0.000	0.00	0.000	0.00	0.000	0.00	0.000
3	0.00	0.001	0.00	0.002	0.00	0.001	0.02	0.002
7	0.00	0.002	0.01	0.001	0.01	0.001	0.03	0.001
10	0.00	0.001	0.01	0.001	0.01	0.000	0.03	0.001
14	0.00	0.001	0.01	0.003	0.01	0.000	0.03	0.001
21	0.00	0.001	0.01	0.001	0.01	0.001	0.03	0.002
28	0.00	0.001	0.01	0.001	0.01	0.000	0.03	0.002

Table A.9 Expansion results of mortar bars using natural sand/ (50% fused silica+ 50% natural sand), Type I Low/ High alkali cement subjected to 3% Sodium chloride solution at 80°C

Days	S-LC at 80°C		FS-LC at 80°C		S-HC at 80°C		FS-HCat 80°C	
	Exp. (%)	Std. Dev.	Exp. (%)	Std. Dev.	Exp. (%)	Std. Dev.	Exp. (%)	Std. Dev.
0	0.00	0.000	0.00	0.000	0.00	0.000	0.00	0.000
3	0.01	0.008	0.46	0.036	0.01	0.007	0.47	0.021
7	0.01	0.009	0.83	0.051	0.01	0.008	0.59	0.025
10	0.01	0.004	1.11	0.067	0.02	0.006	0.75	0.023
14	0.01	0.010	1.42	0.066	0.02	0.006	1.00	0.021
21	0.00	0.008	1.95	0.087	0.01	0.003	1.39	0.035
28	0.01	0.008			0.02	0.006	1.68	0.036

Table A.10 Expansion results of mortar bars using natural sand/ (50% fused silica+ 50% natural sand), Type I Low/ High alkali cement subjected to 3% Calcium chloride solution at 80°C

Days	S-LC at 80°C		FS-LC at 80°C		S-HC at 80°C		FS-HCat 80°C	
	Exp. (%)	Std. Dev.	Exp. (%)	Std. Dev.	Exp. (%)	Std. Dev.	Exp. (%)	Std. Dev.
0	0.00	0.000	0.00	0.000	0.00	0.000	0.00	0.000
3	0.02	0.002	0.02	0.005	0.01	0.002	0.03	0.002
7	0.01	0.002	0.01	0.002	0.01	0.006	0.03	0.002
10	0.02	0.001	0.02	0.001	0.01	0.007	0.04	0.001
14	0.02	0.005	0.02	0.001	0.01	0.005	0.04	0.004
21	0.02	0.001	0.02	0.003	0.02	0.006	0.04	0.002
28	0.02	0.001	0.02	0.002	0.02	0.006	0.04	0.001

Table A.11 Expansion results of mortar bars using natural sand/ (50% fused silica+ 50% natural sand), Type I Low/ High alkali cement subjected to 3% Potassium chloride solution at 80°C

Days	S-LC at 80°C		FS-LC at 80°C		S-HC at 80°C		FS-HCat 80°C	
	Exp. (%)	Std. Dev.	Exp. (%)	Std. Dev.	Exp. (%)	Std. Dev.	Exp. (%)	Std. Dev.
0	0.00	0.000	0.00	0.000	0.00	0.000	0.00	0.000
3	0.01	0.001	0.17	0.004	0.01	0.003	0.24	0.013
7	0.02	0.002	0.39	0.012	0.02	0.002	0.41	0.018
10	0.02	0.001	0.51	0.015	0.02	0.006	0.51	0.018
14	0.02	0.001	0.70	0.007	0.02	0.002	0.67	0.034
21	0.02	0.001	0.93	0.007	0.02	0.008	0.91	0.022
28	0.02	0.001	1.08	0.020	0.03	0.008	1.06	0.021

Table A.12 Expansion results of mortar bars using natural sand/ (50% fused silica+ 50% natural sand), Type I Low/ High alkali cement subjected to 1N Sodium hydroxide solution at 80°C

Days	S-LC at 80°C		FS-LC at 80°C		S-HC at 80°C		FS-HC at 80°C	
	Exp. (%)	Std. Dev.	Exp. (%)	Std. Dev.	Exp. (%)	Std. Dev.	Exp. (%)	Std. Dev.
0	0.00	0.000	0.00	0.000	0.00	0.000	0.00	0.000
3	0.01	0.001	0.53	0.012	0.01	0.002	0.46	0.012
7	0.02	0.001	0.93	0.011	0.02	0.003	0.85	0.033
10	0.02	0.001	1.04	0.006	0.03	0.004	0.97	0.028
14	0.03	0.001	1.18	0.008	0.04	0.003	1.15	0.048
21	0.04	0.005	1.37	0.011	0.06	0.003	1.38	0.046
28	0.05	0.005	1.56	0.122	0.08	0.002	1.51	0.057

Table A.13 Expansion results of mortar bars using natural sand/ (50% fused silica+ 50% natural sand), Type I Low/ High alkali cement subjected to 0.5N Sodium hydroxide and 3 % Sodium chloride blended solution at 80°C

Days	S-LC at 80°C		FS-LC at 80°C		S-HC at 80°C		FS-HC at 80°C	
	Exp. (%)	Std. Dev.	Exp. (%)	Std. Dev.	Exp. (%)	Std. Dev.	Exp. (%)	Std. Dev.
0	0.00	0.000	0.00	0.000	0.00	0.000	0.00	0.000
3	0.00	0.000	0.59	0.040	0.02	0.003	0.53	0.012
7	0.00	0.001	0.90	0.057	0.03	0.003	0.78	0.016
10	0.00	0.006	1.09	0.060	0.03	0.002	0.99	0.021
14	0.00	0.001	1.22	0.055	0.03	0.003	1.13	0.020
21	0.01	0.006	1.47	0.055	0.04	0.002	1.42	0.037
28	0.01	0.005	1.55	0.063	0.06	0.028	1.54	0.028

Table A.14 Expansion results of mortar bars using natural sand/ (50% fused silica+ 50% natural sand), Type I Low/ High alkali cement subjected to 3% Magnesium chloride solution at 80°C

Days	S-LC at 80°C		FS-LC at 80°C		S-HC at 80°C		FS-HC at 80°C	
	Exp. (%)	Std. Dev.	Exp. (%)	Std. Dev.	Exp. (%)	Std. Dev.	Exp. (%)	Std. Dev.
0	0.00	0.000	0.00	0.000	0.00	0.000	0.00	0.000
3	0.02	0.007	0.02	0.009	0.01	0.002	0.04	0.001
7	0.02	0.002	0.02	0.008	0.02	0.002	0.05	0.004
10	0.02	0.004	0.02	0.008	0.02	0.001	0.05	0.001
14	0.03	0.003	0.02	0.006	0.02	0.001	0.04	0.002
21	0.02	0.002	0.01	0.004	0.02	0.002	0.04	0.002
28	0.02	0.004	0.01	0.006	0.02	0.001	0.04	0.002

Table A.15 ICP Test Results

	De-ionized water		3% NaCl		3% $CaCl_2$		3% KCl	
	Std. solution	Water soluble hydroxides	Std. solution	Water soluble hydroxides	Std. solution	Water soluble hydroxides	Std. solution	Water soluble hydroxides
Na	62.04	67.83	548.04	557.14	68.56	68.99	67.43	68.46
K	10.21	16.61	11.01	17.35	11.68	16.99	426.79	418.67
Ca	0.00	16.28	0.00	19.59	267.33	260.65	0.00	19.60
Mg	0.00	0.06	0.00	0.00	1.08	0.12	0.00	0.01
SO_4	3.067	3.416	3.105	3.727	3.486	3.438	3.401	3.662
Cl	23.05	25.69	521.41	528.08	549.03	517.26	426.42	416.42

	1N NaOH		0.5N NaOH+ 3% NaCl		3% MgCl2	
	Std. solution	Water soluble hydroxides	Std. solution	Water soluble hydroxides	Std. solution	Water soluble hydroxides
Na	965.09	707.70	981.59	979.48	67.54	71.19
K	10.75	17.46	10.81	17.76	11.92	17.92
Ca	0.00	0.43	0.00	0.68	5.01	147.97
Mg	0.00	0.00	0.13	0.03	143.83	0.01
SO_4	2.964	5.348	2.907	5.556	3.142	3.340
Cl	24.52	26.91	513.43	527.17	321.37	301.71

Note: Concentration of solution is in millimoles/lit

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